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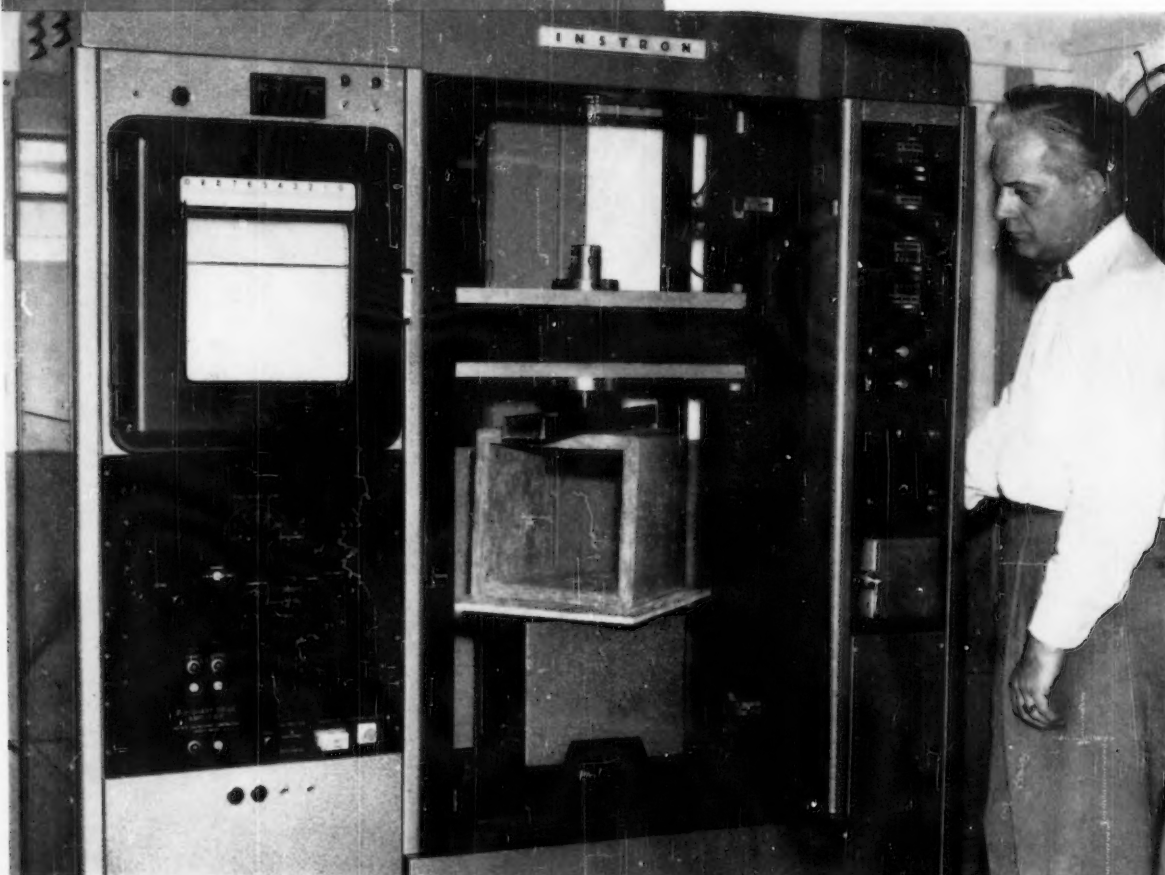
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ASTM BULLETIN

JANUARY 1958

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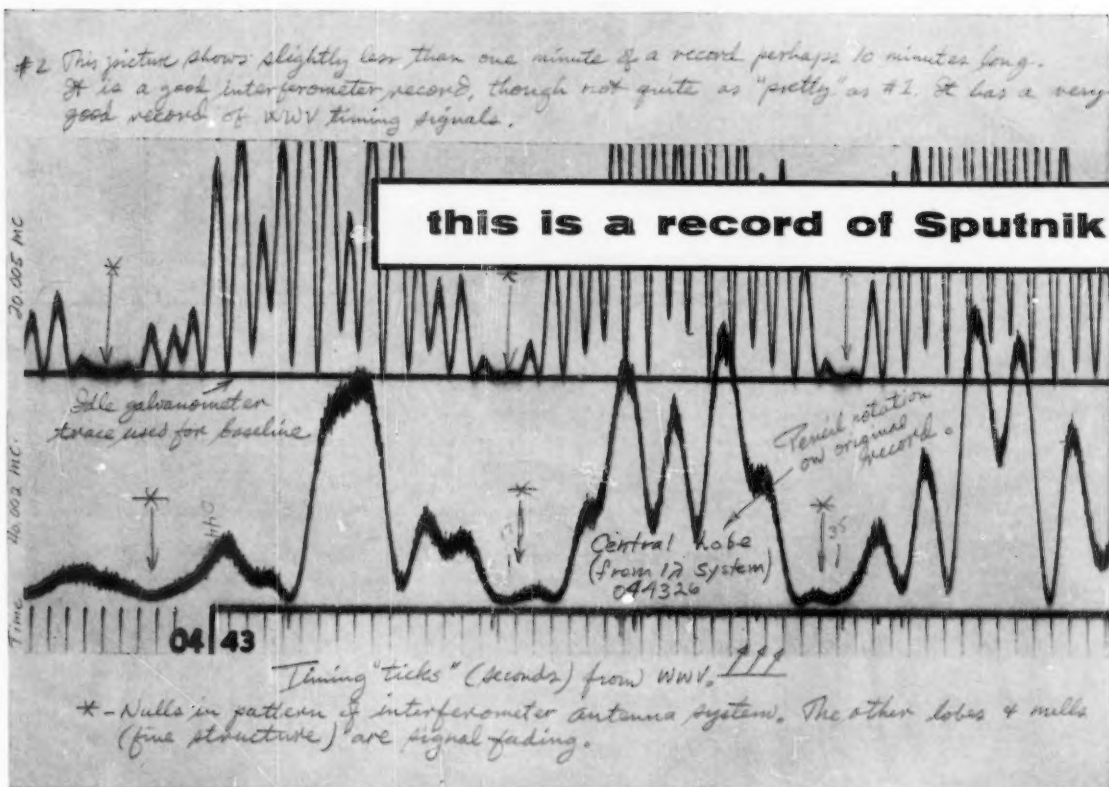
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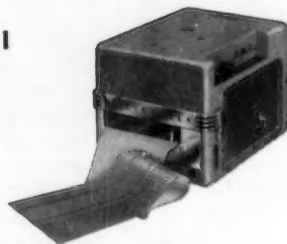


The Visicorder has charted the orbit of Sputnik I

A Model 906 Honeywell Visicorder Oscillograph wrote this record of the signals from Sputnik I for the Department of Electrical Engineering at the University of Illinois at Urbana. The marginal notes are those of Edgar Hayden, the research associate who took the record.

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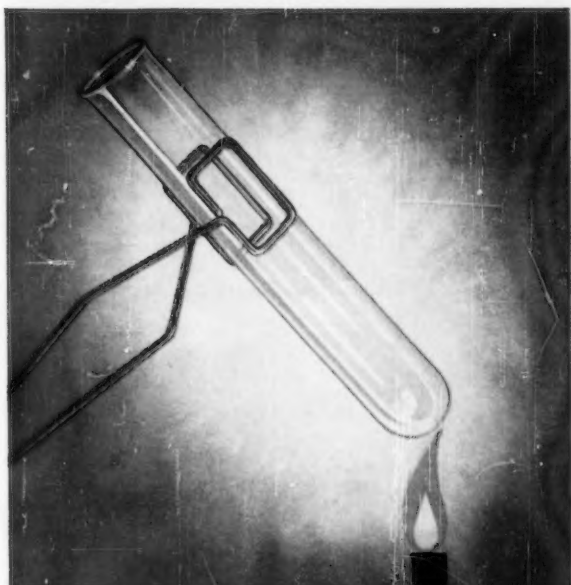
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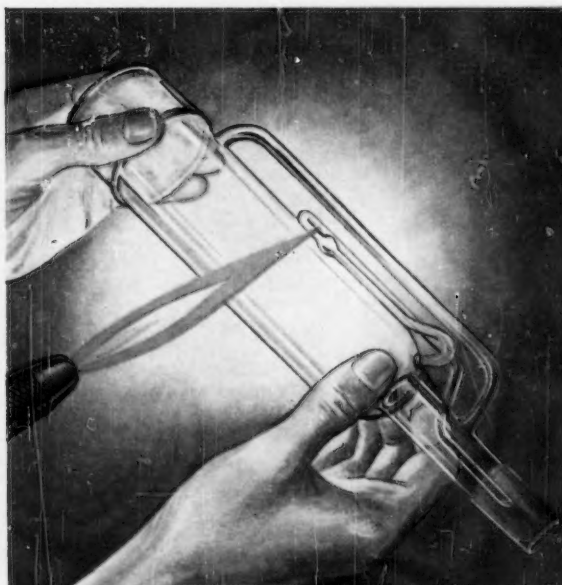
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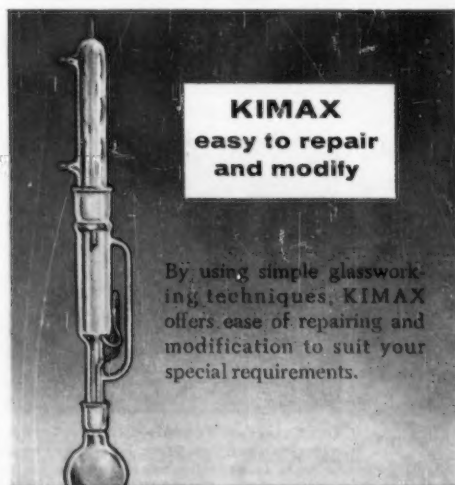
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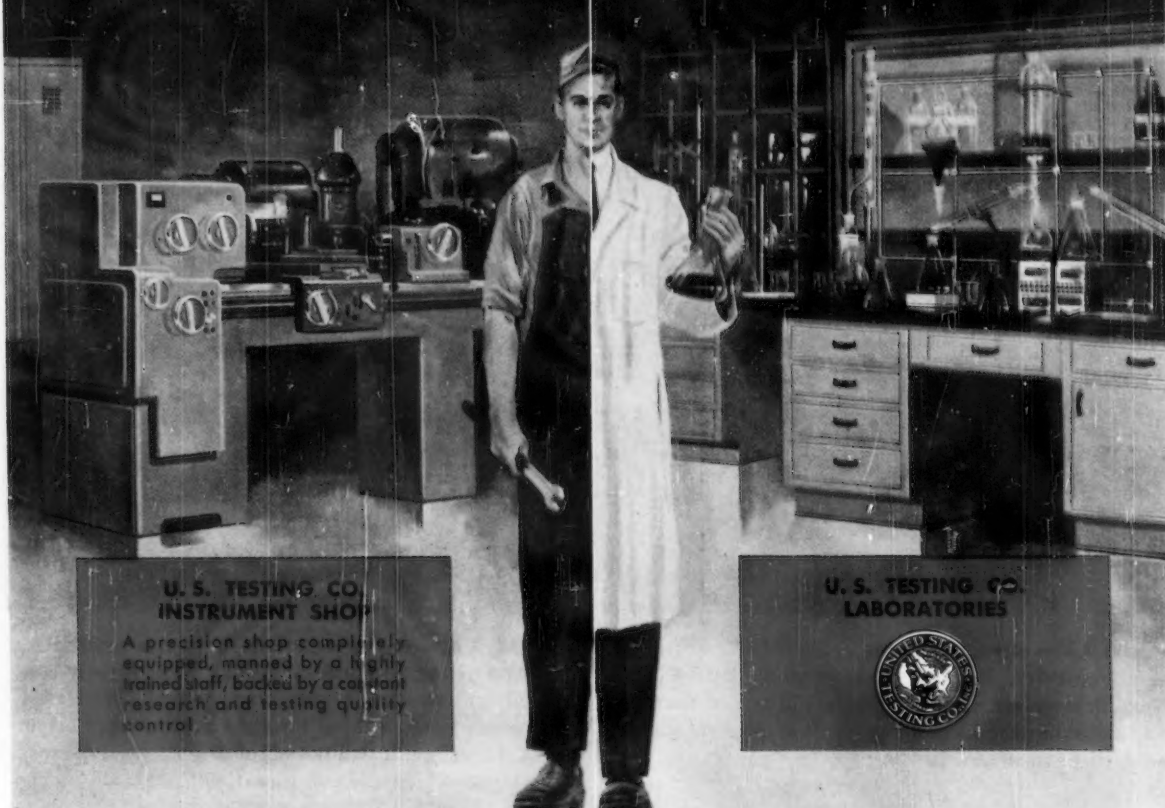
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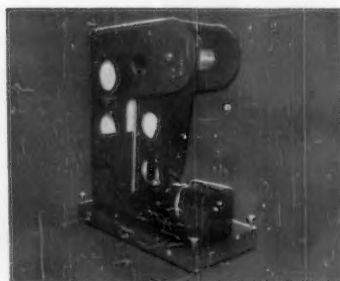


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ASTM in '57**RESEARCH + ENGINEERING KNOW HOW = STANDARDS**

ASTM is a research Society. Indeed it has been said that virtually all of the work that goes on in ASTM technical committees is research, the final result of which is a standard. It is well recognized that adequate specifications cannot be written by pulling figures out of the air, so to speak. The specification levels to be soundly based depend upon exhaustive and careful research both into the properties of materials and their evaluation, as well as an investigation into the application of the materials for which the specifications apply.

While these investigations are not always called research and a definition of research will not be attempted here, it is evident from any comprehensive review of work in ASTM that research, considered broadly, is the keynote for most of the activities. Evidence of the Society's many contributions to research and to the promotion of knowledge of engineering materials may be found in the expanding list of ASTM publications derived from symposia and technical sessions at the Annual Meetings and other national meetings, including Committee Week, as well as the numerous technical sessions and symposia sponsored by individual technical committees at their meetings.

1957 Committee Week and Annual Meeting . . .

During Committee Week, Committee C-16 on Thermal Insulation, sponsored a symposium comprising four papers covering various methods for evaluating thermal conductivity, and one of the effect of moisture on thermal conductivity. Other papers covered testing

of insulation for underground piping and stress-corrosion cracking of insulated stainless steels. The papers in this symposium have been published.

At the Annual Meeting, technical sessions, symposia, and memorial lectures covered industrial water, high-temperature alloys, soils, large fatigue machines, concrete, gases in metals, analysis for trace elements, dissolved oxygen in water, and radiation effects on materials. The radiation effects symposium, jointly sponsored by the Atomic Industrial Forum, was the second in a series of symposia on this subject which are being continued annually under the joint sponsorship.

During 1957 many of the 200 papers from the Second Pacific Area National Meeting in September, 1956, have been published. These papers add to those from the regular meetings of the Society. The success of the Pacific Area Meeting led to the decision to hold a national meeting on the West Coast every third year. The next one is scheduled for the fall of 1959 in San Francisco.

It is evident from attending these symposia and technical sessions and from reading the papers in the *Proceedings*, in the *BULLETIN*, and in *Special Technical Publications*, that research is the backbone of the Society's work. Without this carefully considered research background, no adequate tests or specifications could be written.

Research in technical committees . . .

While some committees prefer to present their more formal technical programs at the Annual Meeting and other National Meetings of the Society, other committees sponsor technical sessions at committee meetings.

Often these technical sessions are informal, the papers may not be published and there is little or no publicity concerning the events. During 1957 a number of committees sponsored this type of program and a few remarks relating to many of them follow:

● Committee B-5 on Copper and Copper Alloys, Cast and Wrought for some time has sponsored a technical session at each meeting, usually with one paper covering some aspect of the committee's work. A recent session included a paper on "Mechanical and Physical Characteristics of Jewelry Bronze 87.5 per cent Strip," by D. E. Trout of Scoville Manufacturing Co. which appears on page 45 of this issue.

● Committee B-8 on Electrodeposited Metallic Coatings often holds technical sessions at its meetings. During Committee Week a highlight of their meeting was a paper on "Chromate Films on Electrodeposited Zinc," by Nagley, Katz, and Proctor of the Bureau of Ships. Color slides were shown indicating the appearance of panels after various periods of exposure aboard ship.

● Committee C-21 on Ceramic White-ware, through its newly organized subcommittee on nuclear applications, assisted in the arrangements for a symposium on nuclear ceramics held at the fall meeting of the American Ceramic Society.

● Committee D-14 on Adhesives usually has at its meetings a technical session with two or three papers related to research on adhesives. Recent topics included discussion of such subjects as orientation of organic molecules as this affects adhesion, and factors contributing to tack or "stickiness" of adhesives.

● Committee D-22 on Methods of Atmospheric Sampling and Analysis, at least one of its meetings during the year, through various individuals on the committee, cooperates with the American Chemical Society in technical sessions and symposia relating to atmospheric analysis.

● Similarly, Committee D-23 on Cellulose, which usually meets in conjunction with the American Chemical Society, contributes to research symposia sponsored by that Society.

● Committee C-1 on Cement has a regular policy that at each meeting one of the subcommittee chairmen will present a technical paper covering the work of the subcommittee and some of its research problems. The chairmen present these papers in turn so that all subjects are covered. While many of these papers are informal, some are later submitted for publication by the Society.

● Committee C-7 on Lime is interested in the research program at MIT on the soundness of lime, and periodic reports on this subject are presented to the committee by Professor Voss.

● Committee C-12 on Mortars has occasional technical sessions and is planning such a session on brick masonry at the February meeting this year.

● Committee C-19 on Structural Sandwich Constructions regularly sponsors a technical session at each meeting, usually in the evening, at which one or two papers are presented. At a recent session there were talks covering adhesives and the use of sandwich constructions in the Convair B-58.

● Committee D-18 on Soils regularly sponsors symposia at the Annual Meeting. A joint symposium with the Mexican Sociedad de Mecanica de Suelos was held in December, at Mexico City.

● Committee D-9 on Electrical Insulating Materials at each meeting sponsors a symposium related to the work of the committee. A number of such symposia on insulating oils have been held and these have been published by the Society. Recently the committee has sponsored symposia concerned with corona testing and with the application of statistics to insulation problems.

● Committee D-20 on Plastics regularly sponsors technical sessions at its meeting at which papers are presented

covering research related to the work of the committee. Recent topics include application of statistics in the testing of polyethylene, and rheological measurements on thermoplastics.

● Committee D-1 on Paints sponsors a technical session at each committee meeting. Topics covered in 1957 included the testing of floor varnishes and exposure testing.

● Committee D-11 on Rubber sponsors a technical session at each meeting, presenting recently papers on dimensional tolerances of wire insulation, a flexing apparatus for ozone testing and the use of statistics for handling evaluation data.

● Committee D-2 on Petroleum Products may have as many as three separate symposia in conjunction with a committee meeting. At recent meetings symposia were held covering viscosity classifications and composition of petroleum.

● Committee D-13 on Textiles, holds a papers session at alternate meetings. Subjects discussed at recent meetings included the vibroscope method for linear density and the use of standards by consumers and test methods for consumer products. Annually the committee sponsors the presentation of the Harold DeWitt Smith Award for outstanding research in the textile field. The recipient at the time of the award presents a technical paper on his research.

New projects established . . .

Any review of the year would naturally include some mention of new areas for research and standardization. A new committee has been approved and the organization effected covering the field of sorptive mineral materials (Committee C-23).

A new committee on flexible barrier materials is in process of organization.

A conference has recommended the organization of a committee on halogenated organic solvents and solvent mixtures, particularly as they apply to solvent cleaning operations both for dip cleaning and vapor degreasing.

An Advisory Committee on Industrial Chemicals has been organized with the objective of guiding the Society's work including any committee expansion in the field of industrial chemicals. Items currently under consideration in this committee include investigation of new standardization projects in such fields as mineral acids and industrial alcohols.

The advisory committee is also studying the feasibility of recommending the establishment of a broad "E" committee covering general problems in chemical analysis and testing of industrial chemicals.

Nuclear activities . . .

The Society continues its efforts toward the establishment of suitable standards for application of materials in nuclear installations either where the effects of radiation are important or where some other property of the material has a bearing on the application. The effects of radiation must be evaluated, but before adequate exposure methods can be written it is necessary to standardize dosimetry, that is, the measurement of the amount of radiation to which specimens are exposed. Problems of dosimetry are complicated by the fact that there are a number of different kinds of radiation—beta rays, gamma rays, X-rays, neutrons, and mixtures of these. Two groups are actively working on the problem of dosimetry, one in Committee E-10 on Radioisotopes and Radiation Effects, which has the responsibility for the society-wide problems, and the other in a joint subcommittee of Committees D-9 and D-20 where the emphasis is primarily on the effect of radiation on organic materials. The work of these two groups is being coordinated.

Technical programs in the districts . . .

1957 has seen an increase of interest in district activities and, particularly, the sponsoring, often jointly with other societies, of technical programs at the district meetings. Technical programs are usually in the form of talks on technical subjects by experts in the field. Some of the subjects covered at District Meetings during 1957 were strength testing of brittle state materials, transistors, industrial water, the science of engineering materials, economic evolution *versus* textile research, and atomic energy. During 1957 more than half of the district technical meetings were joint with other technical and professional societies.

National and international cooperation . . .

During the year the Society continued its extensive participation in the co-

ordination of standards through the activities of the American Standards Assn. Many of the ASTM committees are represented on Sectional Committees of the ASA. About one-third of all the standards approved as American Standards are those developed by ASTM.

ASTM committees have also continued active participation with committees of the International Organization for Standardization, providing the American Groups to the ISO under the auspices of the American Standards Assn., the U. S. member of the ISO.

Friendly cooperation with soils engineers in Mexico was evident in the recent joint meeting of Committee D-18 on Soils and the Mexican Society of Soil Mechanics held in Mexico City. (A full report of these meetings is on page 21.)

Headquarters was host during 1957 to a number of visitors from abroad who wanted to learn more about U. S. practice in science, engineering, and standards related to engineering materials. These groups included a European Productivity Agency Mission of 30 members from eight European

countries, which visited Headquarters in November; the Philippine Industry Standardization Study Team of eight members which visited Headquarters in October; the executives of Japanese steelmaking firms and representatives from the Taiwan Cement Corp. visited in March and April. Also, representatives from the Federal Commission for Standardization for Yugoslavia, visited Headquarters in October.

In addition to all these direct personal interests in ASTM on the part of technical people from abroad, there also were a large number of inquiries about ASTM standards and publications handled by the Headquarters staff. Not only are there members of the Society throughout the world, as evidenced by the geographic listing in the Year Book, but there are many companies and organizations from abroad, not members of the Society, who purchase the Society's publications and write in for information.

There is no doubt that these international activities of the Society add materially in the assistance by this country to many of the underdeveloped areas throughout the world.

a period of use it is found that the alternate materials are adequate for the intended use and continue to be specified. A case in point is the emergency specification for stainless steels (A 421), developed during the Korean war. It has been proved since that the material was quite adequate and it has now become the basis for standard steels for industry.

The chemical industry has special requirements for materials of construction and at the request of the Chemical Industry Advisory Board, advisory to the American Standards Assn., there has been developed a new specification covering welded large outside diameter, light wall austenitic chromium-nickel alloy steel pipe for corrosive or high-temperature service (A 409). This specification will be of special value to the chemical industry in applications requiring the high corrosion resistance and thermal stability of this material.

The special properties of wrought iron as fabricated into welded pipe are covered in a new specification for this material developed during 1957 (A 419).

Turning to nonferrous metals, one

of the large potential uses for aluminum is in electrical conductors. To meet this need a new specification has been developed for conductor-grade aluminum (B 314). Another similar specification was prepared covering aluminum alloy bus bars (B 317).

The use of these materials will supplement the limited supplies of copper in the continuing expansion of power transmission and other conductor needs.

A draft of a monograph on electroless nickel has been circulated for review within Committee B-8 on Electrodeposited Metallic Coatings. Individual chapters concerning properties, production, and testing of electroless nickel had been prepared by members of the committee. Plans are to publish the monograph in 1958.

Nuclear program spurs rare metals specifications

Surveys have indicated that there are industry needs for specifications covering beryllium, columbium, lithium, tantalum, thallium, thorium, uranium, zirconium, and hafnium. Under active development are specifications for beryllium, zirconium, hafnium, and lithium. Development of specifications for the others will be taken up as needed.

Metallography

In the metallographic examination of metals, problems arise in the reporting of results because of conflicting nomenclature to designate alloy phases. As a contribution toward improving this situation, P. A. Beck of the University of Illinois presented a paper at the 1957 Annual Meeting in which a number of suggested improvements were made. This paper which appeared in the December 1957 BULLETIN is being reprinted and given wide distribution to solicit comments toward establishment of improved nomenclature for metallography.

Nondestructive testing

One of the very useful techniques for nondestructive testing involves the use of ultrasonics. However, in order that ultrasonic nondestructive tests may be meaningful it is desirable to establish methods for standardizing the sensitivity of ultrasonic test equipment. Progress has been reported in the use of a standardizing scheme based on the use of steel balls immersed in a liquid.

Standardization activities . . . Metals

With the present emphasis on heavy construction in connection with the highway program and such large projects as the St. Lawrence Seaway, it is fitting that during 1957 two new specifications were issued covering wire strand for prestressed concrete (A 416) and special large-size deformed reinforcing bars (A 408). Another significant completed project was the inclusion in specification A 120 of a section covering steels made by the new basic oxygen process. Similar additions are in preparation covering this type of steel for concrete reinforcement, structural steel, and other steel products.

The steel committee has completed the development of a new specification covering flange and firebox quality steel sheets (A 414) after six years of effort. This specification was originally requested by the Boiler and Pressure Vessel Committee of ASME, but basic disagreements on methods for sampling in determining mechanical properties were such that at one time the project was abandoned.

Sometimes in an emergency, to solve problems of materials shortages, a substitute material is specified. After

A special feature of the year in non-destructive testing was the Symposium on Non-Destructive Tests in the Nuclear Energy Field, held in Chicago in April. The symposium was jointly sponsored by the American Institute of Chemical Engineers, the American Nuclear Society, the Society for Nondestructive Testing, the Atomic Industrial Forum, and ASTM. The proceedings, consisting of some 40 papers with discussion, are being published by ASTM as a *Special Technical Publication*.

Fatigue

The problems of evaluating the resistance of various materials to failure in fatigue—that is, application of cyclic stress—have been under intensive investigation in ASTM for a long time. Much of the experimental data reported in the literature is based on tests of small specimens. The question of whether these data apply in predicting fatigue strength of large specimens remains to be settled. To provide a basis for further consideration of this difficult subject, a symposium on large fatigue testing machines and their results was held at the Annual Meeting.

Another useful contribution of the Society in the fatigue field was the publication in 1957 of references on fatigue covering the previous year. Some 350 references are included in this publication (*STP 9H*).

Also published during the year was the Symposium on Fatigue of Aircraft Structures (*STP 203*), which was presented at the West Coast Meeting in 1956.

Because of the difficulty in analyzing fatigue data there has been under development for some time "A Guide for Fatigue Testing and Statistical Analysis of Fatigue Data." A tentative draft of this publication is being considered by the committee for possible publication by the Society in the near future.

Corrosion

The Advisory Committee on Corrosion continues to supervise the atmospheric exposure test sites in various parts of the country. Participating in the special test program are a number of ASTM committees, mostly in the metals field. The following is a summary of activities in several ASTM committees including both exposure programs and laboratory investigations.

- Committee A-5 on Corrosion of Iron and Steel is preparing a number of

specimens of flat, bent, and round shapes of coated carbon steel, low-alloy steels, and nodular iron. The specimens which are coated with hot-dip zinc, aluminum alloy, electrodeposited zinc, and sprayed aluminum will be exposed at Newark, N. J., and Kure Beach, N. C.

Data on the atmospheric exposures of wire and wire products in progress for 20 years at eleven test sites were reported in the 1957 Annual Report of Committee A-5. Data from the 28-year inspection of hardware exposed at State College, Pa., were also included.

- Committee A-7 on Malleable-Iron Castings is assembling specimens of malleable iron, pearlitic malleable iron, and nodular iron, together with three types of steel, to be exposed at five test sites. This investigation is expected to provide some reliable information on the corrosion rates of malleable and pearlitic iron.

- Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys plans to expose a large number of stainless steel specimens at three test sites. Specimens will include tubing, sheets, and coiled springs, both arc and spot welded. The test program is planned to obtain comparative data on the performance of commercially produced flat rolled products in typical atmospheric environments.

- Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys is undertaking a large program to expose at several locations 60 specimens each of 74 different alloys. The committee is also preparing couples of magnesium with a variety of other metals, for exposure at three test sites, to gain information on corrosion rates of most of the commercial non-ferrous alloys.

- Committee B-8 on Electrodeposited Metallic Coatings has completed exposure of a large number of chromate-treated chromium plated panels at three different locations. Data obtained are expected to provide definite information on corrosion resistance afforded by chromate treatment. The committee also has in progress a number of other exposures of copper-nickel-chromium and nickel-chromium panels, as well as chromium-plated items which are being examined periodically.

- In 1955, Committee C-19 on Structural Sandwich Constructions initiated exposure of a number of sandwich panels at State College, Pa., and Kure

Beach, N. C. The specimens were removed for examination in 1957 and a new series of 40 specimens was placed on exposure at the same sites. Specimens from this group will be removed annually for inspection.

- Committee D-15 on Engine Antifreezes has long been concerned with the measurement of corrosive effects of engine antifreezes. The committee has designed a special mechanical bench equipment corrosion test designed to provide results useful in indicating the relative corrosiveness of the antifreeze in an engine. Twenty of these units, including a special reservoir equivalent in size and material to an engine block, are now being manufactured for the first interlaboratory test program for evaluating antifreeze corrosivity.

- Committee D-10 on Shipping Containers is concerned with the corrosive staining and tarnishing characteristics of cushioning materials when in contact with packaged articles, and is devising a test to be conducted in a conditioned atmosphere of 100 F and 90-95 per cent RH for a period of 14 days.

- The ALCA-ASTM Joint Committee on Leather is developing a test to evaluate the corrosive effect of leather in contact with metal.

- Committee D-20 on Plastics also is devising a similar test for plastics.

Effect of temperatures

The ASTM-ASME Joint Committee on Effect of Temperature on the Properties of Metals continued its active program of collecting and publishing useful compilations of data relating to the high-temperature properties of metals. Following are reports of several projects completed or in progress:

Project DP-5. Survey of Relaxation Data.—This project was completed in 1957 with the publication of "Relaxation Properties of Steels and Super Strength Alloys at Elevated Temperatures," *STP 187* (1956).

Project DP-8. Survey of Medium-Carbon, High-Chromium and Plain Carbon Steels.—Two-thirds of the project has been completed with the publication of "Elevated Temperature Properties of Carbon Steels," *STP 180* (1955), and "Elevated Temperature Properties of Wrought Medium-Carbon Alloy Steels," *STP 199* (1957).

Project DP-6. Survey of Strength of Weldments.—Final manuscript covering the evaluation of some 200 data sheets has been submitted for publication. It is scheduled for publication in January, 1958 as STP 226.

Project DP-12. Survey of Test Facilities.—The results of this survey are complete and available in mimeograph form.

Elevated Temperature Properties of Cast Iron.—This work is being carried out at the Southern Research Inst., and a final report will be presented at the ASTM Annual Meeting in June, 1958.

Calibration Specimen.—This project will provide calibration specimens for creep-rupture testing. The National Bureau of Standards will act as distributing agent.



Testing for Flatness. *Third Prize General Photographs, black & white*—Tenth ASTM Photographic Exhibit. W. M. Pedziwiatr, Crane Co., Chicago, Ill.

Nonmetallic Construction Materials

Cement

Extension of inspection services of the Cement Reference Laboratory to include concrete testing facilities was an important recent decision of Committee C-1 on Cement. Means of implementing this expanded service are now being planned. Several active projects on portland blast-furnace slag cement cover heat of hydration, sulfate resistance, and ignition loss. Further refinements in chemical analysis procedures were accomplished. A highlight of the year was a very successful meeting in Mexico arranged by the Mexican presentation on the committee.

Chemical mortars

Committee C-3 on Chemical-Resistant Mortars has completed three recommended practices to round out complete coverage of the four general types of chemical-resistant mortars, namely, sulfur, hydraulic cement, silicate, and resin-type mortars. These recommended practices serve as a very useful guide for storage, mixing, and use of these mortars, as well as for defining good practice in application. Additional test methods were completed and accepted covering working and initial setting time and flexural strength, both pertaining to silicate type mortars; and absorption and apparent porosity, pertaining to silicate resin and sulfur type mortars.

Clay pipe

With the trend toward longer pipe lengths, Committee C-4 on Clay Pipe reviewed the specifications for the purpose of revising the dimensional standards to provide heavier pipe where needed, particularly by increasing the thickness of shoulders.

Lime

Committee C-7 on Lime added to its list of standards covering lime a new method for testing quicklime and hydrated lime for use in the treatment of waste acid solution. Extensive revisions were made in the methods of sampling, inspection, packing, and marking of quicklime and lime products (C 50). The study leading to the preparation of a more suitable soundness test method for lime was accelerated with particular attention being given to a survey of available research information. The use of pozzolanic materials as an admixture with lime is the objective of continuing cooperative tests.

Concrete

Recognition of the increasing use of lightweight and cellular type concrete and the special testing problems which this material poses resulted in the establishment in Committee C-9 on Concrete and Concrete Aggregates of a new group on this subject. Evidence

of continuing interest in pozzolanic material is the new specification for raw or calcined natural pozzolans for use as an admixture in portland-cement concrete. This work also is related to the study of alkali-aggregate reactions as this affects the reduction of expansion of concrete. This has proved to be a serious problem with certain combinations of materials. Abrasion resistance of concrete—a significant property for certain applications—can now be measured by means of a proposed method of test accepted at the fall meeting. Additional proposed test methods nearly completed cover the measurement of static modulus of elasticity and Poisson's ratio in compression of molded concrete cylinders. Another new item is a specification for cotton mats for curing. Committee C-9 cooperated with Committee C-1 in a Joint Meeting in Mexico in December.

Gypsum

The work of Committee C-11 on Gypsum during the year resulted in further refinements in its specifications and in the development of specifications covering accessories used with gypsum and gypsum products; one of these was joint tape and cement.

Mortars

Reinforced masonry has become a standard type of construction in the masonry field. This involves the use of a grout, and Committee C-12 on Mortars for Unit Masonry has pre-

sented to the Society a specification for aggregates for this use. Air entraining and grading requirements in masonry mortar received attention, and revisions in existing specifications are planned.

Concrete pipe

The assignment of jurisdiction for concrete drain tile to Committee C-13 on Concrete Pipe has now led to the development of a separate specification for concrete drain tile which was approved at the last committee meeting. This specification covers three classes of nonreinforced concrete drain tile with internal diameters from 4 in. to 24 in. The committee has studied the application of rubber gaskets in concrete pipe joints and has drafted a specification for such joints of rubber composition.

Glass

The need for closer control and more details of procedure in testing glass containers was expressed by Committee C-14 on Glass and Glass Products, and to this end a new series of round-robin tests has been inaugurated to establish data. The need for a standard method of sieve analysis for raw materials used in glassmaking will also be filled when a proposed standard developed by the committee is accepted. Cooperative tests are under way on the calibration of the sieves, including an evaluation of the precision to be expected.

Masonry units

The drying shrinkage of concrete block is a current problem of Committee C-15 on Manufactured Masonry Units. Two proposed test methods have been reviewed, one a modification of a British method and the other the Menzel relative-humidity method. Industrial floor brick, now covered in a new specification, is used in many industries and is designed to cover diverse needs. Therefore, the requirements in the specification are correspondingly complex.

Thermal insulation

One is impressed with the diversity of products covered when reviewing the many standards developed by Committee C-16 on Thermal Insulating Materials. The number of products involved is still increasing, with the recent consideration of poured-in-place materials. A critical review of the important physical characteristics of insulating materials, particularly of the block and pipe type, has been under

way during the year toward improving existing standards. Density, thermal conductance, compressive and flexural strength, pH value, and absorptivity were found to be the most significant properties. The development of recommended practices was continued, on the basis that they are valuable supplements to the standard specifications. The latest recommended practice accepted is one for prefabricated and field fabricated insulation coverings for fittings, flanges, and valves.

Asbestos-cement

Continuing projects of Committee C-17 on Asbestos Cement Products include development of an evaluation test for handleability, and simulated service types of test including a pendulum impact test. A proposed specification for asbestos-cement sewer pipe is currently being considered.

Building stones

Two specifications for natural building stones accepted by the Society are the first of such standards developed by Committee C-18 on Natural Building Stones. In fact, these specifications represent the first which have been published as national standards embodying physical requirements essential for most building construction where engineering is required. A specification for roofing slate covers natural slate shingles as commonly used on sloping roofs and also square or rectangular tiles for flat roof coverings. A second specification covering structural granite was accepted for publication as information only and is not an official ASTM standard. This specification covers the selection, sampling, and testing of granite for specific construction uses. Other groups in the committee have continued to work on proposed specifications for marble, sandstone, and limestone.

Structural sandwich

Additional coverage of test methods for both core materials and sandwich constructions were developed by Committee C-19 on Structural Sandwich Constructions. New tentative methods were accepted by the Society in June covering flatwise flexure strength of sandwich construction and shear fatigue of sandwich core materials. Since June, the committee has approved proposed methods for determining impact resistance of sandwich core materials and is now considering a method for determination of fluid flow in honeycomb cores.

Acoustical materials

The measurement of the sound absorption properties of acoustical materials continues to be of prime concern to Committee C-20 on Acoustical Materials. The latest draft of the comprehensive reverberation-chamber test method was reviewed and accepted by the responsible subcommittee at the fall meeting of the committee. Test procedures of a smaller-scale nature also received attention involving the use of the horn coupler with the impedance tube method (C 384) and the box method for measuring sound absorption.

The establishment of flame resistance classifications of acoustical materials is a current project and a proposed method based on a modification of the method included in Federal Specification SS-A-118g is being prepared. The committee approved a proposed specification for adhesives for the installation of acoustical tile after some difficulty in establishing a suitable aging test procedure. It was agreed that this proposed specification should be issued under the jurisdiction of Committee D-14 on Adhesives.

Sorptive materials

A new field of materials received attention during the year as a result of the organization of Committee C-23 on Sorptive Mineral Materials. The Subcommittee on Test Methods has reviewed the literature and existing industry methods to provide background information for the development of ASTM standard methods.

Paving materials

The activity of Committee D-4 on Road and Paving Materials consisted primarily of refinement of existing standards resulting from cooperative test programs. Further coverage of bituminous mixtures was provided by a new tentative method of test for moisture or volatile distillates in bituminous mixtures (D 1461). The density of soil in place can now be determined by a newly approved method using the sand cone procedure.

Wood

The outstanding activity sponsored by Committee D-7 on Wood continued to be the comprehensive research program on wood poles. The last series of tests involving treated poles is planned for completion by early 1958. A supplementary program involving the testing of treated poles to evaluate the effect on strength of such treatment was inaugurated during 1957. The

committee is developing an accelerated laboratory method of testing the natural decay resistance of woods and a test method for determining the effectiveness of wood preservatives in stakes exposed in field plots. A new test method for determining the hardness of modified woods was accepted by the Society as an additional procedure in the specification for modified wood (D 1324).

Waterproofing

The scope of Committee D-8 on Bituminous Waterproofing and Roofing Materials was expanded in 1957 to include bituminized-fiber pipe, and corresponding expansion of the committee was authorized to develop standards in this field.

Soils

A complete revision of the "Procedures for Testing Soils" was accomplished by Committee D-18 on Soils for Engineering Purposes, with publication now in process. This publication includes a number of new test procedures as well as proposed methods of sampling of soils. An extensive list of terms relating to soil mechanics and foundation engineering was completed in cooperation with the American Society of Civil Engineers. These terms will be recommended in a revision of ASTM Standard D 653. A high point in the activity of the committee during the year was a joint meeting with the Mexican Society of Soil Mechanics in Mexico City during the week of December 9. Fifteen papers were presented, with contributions from members of both organizations.

Building tests

A new project established by Committee E-6 on Methods of Testing Building Constructions covers the testing of window assemblies. Collection of information on the phases of window testing is the first objective. The committee also initiated another new project to develop a test method for use with vapor barriers beneath concrete slabs-on-ground. A proposed method originally prepared by the Forest Products Laboratory is being reviewed. Progress was reported on the development of test methods for masonry units. The committee met in October at the Building Research Center at the National Research Council of Canada in Ottawa.

Chemical Products—Fuels—Water

Paint

The psychology of color has been much publicized recently and there is increasing application of color in industry. Machine shops and assembly lines that were once drab and dull are now bright and cheerful. The refineries are even painting in colorful pastels their large fuel tanks. As evidence of this increasing interest in color, Committee D-1 on Paints established three new tentative methods for color measurements: a test for color-difference using the General Electric Spectrophotometer (D 1482); a test for two-parameter 60-deg specular gloss (D 1471); and a test for color with the "Color Eye" (D 1495). Other recently developed tentatives recommended by the paint committee cover a specification for soy bean oil, a method for sampling oil and fatty acids, tests for density of paints, for indentation hardness of coatings, and for oil absorption of pigments.

Rubber

The types and varieties of synthetic rubber and elastomers are increasing at a rapid rate and in order to deal with the problems encountered in keeping track of such a variety of products, Committee D-11 on Rubber has established a procedure for assigning numbers to new polymers and has authorized a special subcommittee to make such assignments. A series of articles has been initiated in the ASTM BULLETIN under the heading "New SBR and SBR Latex Numbers" in which the new numbers assigned will be published. Once a year, it is planned to incorporate the new numbers and descriptions in ASTM Recommended Practices for SBR and SBR and BR Latexes (D 1419 and D1420). The first such announcements appeared in the September, 1957 BULLETIN and the second in the December issue.

As part of its responsibility in the former synthetic rubber program of the Rubber Reserve, now industry sponsored, the committee has established a new tentative method for sampling and sample preparation of synthetic elastomers (solid styrene-butadiene copolymers) (D 1485). Two other new standards are the test for indentation of hard rubber by type D durometer (D 1484) and the method for strain testing of vulcanized rubber.

The Rubber Committee is planning a symposium on the Effect of Ozone at

the ASTM Committee Week meetings in February.

Textiles

How can one tell whether one blanket is warmer than another, one coat or suit is warmer than another? When we shop for these items, there is no objective way of determining this. Committee D-13 on Textiles has made a definite contribution toward this problem in the establishment of a test method for measuring the thermal transmittance of textiles using a guarded hot-plate method (D 1518). While the test will not be of use to the consumer when he purchases a blanket or a coat, it may help the manufacturers of these items to select the better material. The textile committee has also developed several other standards covering items of direct interest to the consumers—tests for tufted pile floor coverings (D 1485), tests for shrinkage in laundering and dimensional restorability of warp knit fabrics (D 1487), and test for dimensional change of knit fabrics (D 1470).

The textile committee has also added to its comprehensive definitions of terms (D 123) a chart covering the classification of fibers used in textile fabrics. The committee also revised two methods dealing with resistance of textiles to microorganisms.

Adhesives

Recognizing the industrial importance both of wood-bonding adhesives and structural metal-bonding adhesives, Committee D-14 on Adhesives established subcommittees on these subjects during 1957. The two groups are to study mechanical properties, durability, metal surface preparation, and quality control methods for metal-bonding adhesives and durability and working properties of wood-bonding adhesives.

Plastics

The widest used plastic—polyethylene—covers such a variety of grades of material and goes into so many different applications that the problem of writing an adequate specification for it has occupied much of the attention of Committee D-20 on Plastics during 1957. The material is produced by high pressure, by low pressure, with special catalysts, having high density and low density, and with a variety of properties for the different grades.

Progress is being made, however, toward the improvement of the present specification for this material (D 1248). One point that has received special attention is the method for measuring the tendency for the material to crack when stressed under certain environmental conditions. The committee has published a method for environmental stress cracking which appeared both in the ASTM BULLETIN and the 1957 edition of the Compilation of ASTM Standards on Plastics. Efforts toward improving this test will continue.

The Joint Committee on Plastic Pipe, organized several years ago in cooperation with the Society of the Plastics Industry, has completed the development of several specifications and tests for plastic pipe which probably will be recommended for publication soon. These cover specifications for acrylonitrile-butadiene-styrene pipe, solvent welded pipe, and polyethylene pipe. Also covered are test methods, both short time and long time, for evaluating burst strength.

Radiation Effects Group Active.—At several meetings during the year, the Subcommittee on Radiation Effects jointly under the plastics and electrical insulation committees, has developed an active program. Projects occupying much of the group's attention cover development of methods for measurement of radiation dose and procedures for radiation exposures to evaluate radiation effects. The subcommittee has also developed an educational program and is sponsoring a series of seminars on radiation effects at committee meetings.

Cellulose

Committee D-23 on Cellulose has cooperated in the establishment of a number of standard samples of cellulose to be used throughout the world as a basis for developing standard cellulose methods. This has been in cooperation with the International Committee on Cellulose Analysis. Specific test methods being evaluated by the committee are alkali solubility tests, and a method for intrinsic viscosity. Other methods being studied include those for determining color, moisture content, chain length distribution, and determination of hydroxyl value of cellulose esters.

Leather

The Joint Committee on Leather jointly sponsored by ASTM and The

American Leather Chemists Assn. published an extensive glossary of terms with its 1957 Annual Report to the Society. Also appearing in the report were methods for measuring the area and width of leather. A number of problems are currently under investigation dealing with the effect of water on leather and surface characteristics and appearance.

Petroleum products

While reciprocating engines will be used to power aircraft for some years to come, the new aircraft that are being built are powered by turbines and jets. Therefore, the current emphasis in Committee D-2 on Petroleum is in developing standards for aviation fuels for these new types of power plant. Evidence of this is the proposed method for thermal stability of aviation turbine fuels, published in 1957. The method is the outcome of cooperative work carried out by the Coordinating Research Council from 1955 to 1957, and it describes a procedure for rating the tendencies of aviation gas turbine fuels to deposit decomposition products in the fuel system components in high performance engines. The committee has also published a proposed specification for aviation turbine fuels.

Thirteen other proposed methods and specifications developed by the committee cover volume and weight tables for benzene, toluene, and *p*-xylene; revisions in the research method for knock test rating to include fuels over 100 octane; method for distillation of petroleum products; and a test for odor of petroleum wax. The petroleum committee also completed the development of a number of new tentatives and revisions covering methods for measuring color of petroleum, tests for ball-bearing greases, for freezing point of aviation fuels, for heat of combustion, for tensile strength of paraffin wax, for needle penetration of petroleum wax, and methods of test for sulfur in petroleum products and liquefied petroleum gas.

Coal

Committee D-5 on Coal has been concerned for some time with proper plans and procedures for coal sampling. An extensive theoretical study of coal sampling, initiated in 1955 following a symposium on the subject in 1954, is continuing. Results thus far have shown that there is good agreement between actual sampling and that which would be predicted by the theoretical methods. A formal report will be prepared this year.

Shipping containers

Committee D-10 on Shipping Containers has had a long history of successful operation in the Society, having been established some 50 years ago. This 50-year history was documented by a report which appeared in the February, 1957 ASTM BULLETIN.

It is well known that tremendous losses can be incurred through damage to merchandise in shipment, and there is no doubt that the committee has contributed immeasurably to minimizing these losses through the establishment of standard methods of evaluating shipping containers. The committee is currently initiating an extensive program for developing tests for interior packing. A number of proposed methods are being evaluated, and further work is being directed toward evaluation of dusting and breakdown, compatibility (corrosion test), and dynamic shock testing of cushioning materials.

Water

Your Most Important Raw Material—Water—was the subject for the 1957 Edgar Marburg Lecture, presented by Everett P. Partridge. Dr. Partridge emphasized the importance of water as an industrial raw material and described some of the industrial achievements in its handling and use. He pointed out the growing importance of ultra-pure water containing no more than 50 parts per billion of total impurity. Ultra-pure water has been the subject for much of the effort in Committee D-19 on Industrial Water. In 1957 the committee recommended the publication of a new tentative test for iron in high-purity water (D 1497) which marks the first published standard of the Society in the field of high-purity water.

Another topic which has occupied much of the time of the committee is that of dissolved oxygen in water, and a symposium on this subject was held at the 1957 Annual Meeting. The proceedings have been published as *STP 219*.

Carbon black

Committee D-24 on Carbon Black, organized in 1956, got off to a good start by recommending the publication of nine new tentative methods for evaluating carbon black. These were published in its Annual Report in 1957.

Electrical and Electronic Materials

Conductors

As mentioned above under Metals, emphasis in the conductor field is toward the use of more aluminum to supplement copper which continues to be in short supply. Specifications for aluminum communication cable have been approved and published. Also a number of standards dealing with copper conductors have been revised and brought up to date.

Resistance materials and contacts

Resistance materials, both for the manufacture of electrical resistors and materials for electrical heating as well as electrical contact materials and thermostat metals, are within the scope of Committee B-4. The committee has approved for publication a recommended practice for dimensional standards for projection welding electrical contacts. This marks the first of such contact dimensional standards to be developed by the committee. It is expected that others will follow, the next one probably for rivet contacts. The work on thermostat metals progressed with the improvement of method B 106 for flexivity testing by extending the thickness range from 15 mils down to 4 mils. The committee has also recommended publication of a group of standards on thermostat metals. It is believed that availability of this collection of standards will help publicize the work of the Society in this field.

A new machine is being designed to test contact reliability. This is a problem that the committee has worked on for a long time and it is certainly one of great importance when it is considered that the failure of a contact or other component in an electrical or electronic system can cause failure of the whole system.

Tube materials and semiconductors

Committee F-1 on Materials for Electron Tubes and Semiconductor Devices has increased its activities during 1957 and has established a new subcommittee on methods of analysis which will assist all the other groups within the committee. One problem of particular interest is that of analyzing cathode nickel, and the group is co-operating with Committees E-2 and E-3 on this and other analytical problems. The committee continues to revise and bring up to date its method for measuring interface impedance characteristics of vacuum tube cathodes

(F 300). The committee has also established new specifications for sheet and strip used in electron tubes.

While efforts continue toward developing standards for the semiconductor metals, germanium and silicon, there are as yet no completed standards for these materials.

Insulation

In the insulation field, Committee D-9 on Electrical Insulation is paying particular attention to problems relating to corona measurement, are tracking resistance of insulation, thermal stability, as well as special problems relating to test methods for printed circuit materials, particularly copper-clad thermosetting laminates. Methods have been published as information on corona measurement and are tracking. The committee is planning a research project in cooperation with NEMA and EIA directed toward solving some of the problems of specimen preparation and electrical measurements on copper-clad laminates.

The committee has been particularly interested in developments of the International Electrotechnical Commission, Technical Committee 15 on Insulation, and has pursued an active program to evaluate some of the proposals from abroad toward the development of international standards for electrical insulation.

Insulated wire and cable

The subcommittee on magnet wire of Committee D-9 continues an active program toward the development of test methods for magnet wire insulation. One problem in particular is that of compatibility of the insulation with refrigerants as used in hermetically-sealed refrigeration units. So far no standards in the field have been recommended for publication.

Efforts of the wire and cable subcommittee of Committee D-11 on Rubber and Rubber-Like Materials are being applied to the development of specifications for silicone rubber insulation; synthetic rubber, heat-resisting, and moisture-resisting insulation.



Metallurgical Sample (Broken Drive Shaft). *Honorable Mention, General, black & white—Tenth ASTM Photographic Exhibit. John Campbell, Aldan, Del. Co., Pa.*

General Testing and Analysis—"E" Committees

One of the many functions of Committee E-1 on Methods of Testing is to consolidate similar methods into general methods applicable to the testing of a number of different materials. One field where this effort has been especially fruitful is in the area of distillation. During 1957 Committee E-1 consolidated distillation apparatus descriptions from six ASTM methods into a single proposed specification for distillation equipment, which has been published as information. The six methods cover gasoline, distillate fuel oils, plant spray oils, aromatic hydrocarbons, and lacquer solvents and diluents. The committee has also revised and brought up to date general methods for verification of testing machines and extensometers, specifications for microchemical apparatus and test for thickness of electrical insulation. This latter test is applicable to the testing of most sheet materials, particularly if they are hard and relatively incompressible under moderate pressures. The committee has approved for publication a new tentative method of test for determining pore diameter and permeability of porous filters for laboratory use (E 128).

Chemical and spectrochemical analysis

Several committees cover various aspects of chemical analysis, including Committee E-2 on Emission Spectroscopy, E-3 on Chemical Analysis of Metals, E-13 on Absorption Spectroscopy, and the Joint Committee on Chemical Analysis by Powder Diffraction Methods. Two of these committees have extensive projects providing data useful to analysts. The Joint Committee is continuing its work in compilation of X-ray Powder Data Cards. In addition to ASTM, sponsors of the Joint Committee are the American Crystallographic Assn., the British Institute of Physics, and the National Assn. of Corrosion Engineers. Another contributor to the project is the Technische Physiche Dienst in the Netherlands.

Committee E-13 also continues its active project in compiling infrared data on IBM index cards. The collection now totals 14,688 cards. These data are quite useful to the analyst in identifying unknown compounds by reference to the cards applying machine sorting techniques. Committee E-13 during 1957 developed and recommended to the Society its first ten-

tative covering definitions of terms and symbols relating to absorption spectroscopy (E 131), thus adding to the many other definitions of terms published by the Society.

In making determinations by emission spectroscopy, the type and character of the electrodes are of great importance. In order to standardize some of the hard-to-define areas, Committee E-2 has developed a recommended practice for graphite electrodes (E 130). This will provide a basis for reference in a number of emission spectrographic methods developed by this committee. The committee has also developed, in cooperation with Committee F-1 on Electron Tube Materials, a new tentative method for spectrochemical analysis of nickel alloys (E 129). The committee is very much concerned with analysis for trace elements and sponsored a symposium on this subject at the Annual Meeting.

Of particular interest to a number of ASTM committees dealing with metals was the Symposium on Gases in Metals, sponsored by Committee E-3 at the Annual Meeting. Gases are usually not wanted in metals as they impair physical properties for many applications. In particular, gases in the materials used for turbine buckets and generator rotor blades are known to have a deleterious effect. Therefore, methods for making this determination are very important. This symposium provides a base line for improvement of methods for gases in metals and establishing new methods.

Another analytical tool covered by an ASTM committee is that of mass spectrometry. Committee E-14 on Mass Spectrometry, in May, held its 5th Annual Meeting at which 62 papers were presented covering the entire field of mass spectrometry. There are no plans for publishing these papers by ASTM although individual papers will probably be submitted for publication in various journals.

Statistics and quality control

Probably no one subject is of more fundamental importance or has a more far-reaching influence in the Society's work than that of statistics, whether it be in the planning of an interlaboratory test program, in the presentation of data, in the analysis of data to determine the relative contribution to the result of the test method, the material, and the operator of the test. Committee E-11 on Quality Control of Materials continues its active program in advising the technical committees of the Society on this subject.

The Manual on Quality Control, prepared by this committee has been reprinted six times and is a standard reference both for ASTM committees and for industry in general. The committee performed a very useful service last year in the compiling and publishing of a survey of sampling plans in ASTM specifications. This survey may well be the first step toward a general improvement in requirements for sampling included in specifications.

The committee is planning a comprehensive program for a symposium on bulk sampling to be held at the 1958 Annual Meeting.

ASTM Standards at Work

ASTM in the Kindergarten

CRAYONS used by preschool children must meet rather exacting standards. For example, they must be able to withstand pressure exerted by children and the heat from hot little hands. They must not be too soft so that globs of smeary color are found throughout playrooms, yet they must maintain an ease of writing without exceptional grittiness.

To insure the quality of their crayons, one major company has had its testing laboratories performing tests which follow ASTM procedures. These include method D 87 for the melting point of paraffin wax; method D 1320 for the tensile strength of paraffin wax; and method D 1321 for the needle penetration of the petroleum wax. On the basis of these tests the crayon company is able to control heat distortion, melting point, and flexural strength of their crayons.

Committee Week in St. Louis

Holaday, Veeder, and Hochwalt to Speak

PROBLEMS on engineering materials for our missile and other defense programs, for research, for industry, and for better living will be studied at about 300 technical committee meetings during the American Society for Testing Materials Committee Week at the Hotel Statler, St. Louis, Feb. 10-14, 1958.

Supplementing the work of the technical committees will be addresses by prominent industry and Government leaders, relating to the work of the Society and industry. William M. Holaday, U. S. Director of Guided Missiles, will be the guest of honor and speaker at a dinner on Wednesday, February 12. He is expected to speak on matters of concern to the defense program.

N. P. Veeder, president of the Granite City Steel Co., Granite City, Ill., will speak at the Steel Industry Luncheon sponsored by ASTM Committee A-1 on Steel, Tuesday noon, February 11.

Carroll A. Hochwalt, vice-president and director of research, Monsanto Chemical Co., St. Louis, will speak at

the Chemical Industry Luncheon to be held on Wednesday noon, February 12.

Two Open Symposia

Two of the technical committees are holding open sessions at which papers will be presented. Committee D-11 on Rubber has arranged a two-session symposium dealing with ozone problems, and Committee C-15 on Manufactured Masonry Units is developing a session on mortar problems. Speakers will be reporting on very late information in many cases and there are no plans to publish the papers in the near future. Therefore, the only way to be sure of getting the information presented is to attend the sessions. Interested visitors will be welcome whether or not they are members of the Society.

Numerous visits to plants and laboratories having unique testing equipment to show are being planned by a local committee. Information on these will be available at the registration desk. Local arrangements for the meeting including the responsibility for the dinner are in charge of a general committee on arrangements headed by A. Carl Weber, Laclede Steel Co.

ration by Committee D-1. In this new tentative a suitable method of test employing the Color Eye is provided.

SHIPPING CONTAINERS

Method of Drop Test for Shipping Containers (D 775-47) (Approved Dec. 13, 1957)

Revision and Reversion to Tentative.—Experience with this method which has been verified by interlaboratory collaborative studies has indicated to Committee D-10 a need for revision to clarify procedure and improve reproducibility of test results.

SOILS FOR ENGINEERING PURPOSES

Tentative Method of Test for the Load-Settlement Relationship of Individual Piles Under Vertical Axle Load (D 1143-50 T) (Approved Dec. 2, 1957)

Revision.—Committee D-18 has made eight changes in this method consisting in the main of more precise statements to correct ambiguities and uncertainties that have arisen in the use of the method.

PLASTICS

Tentative Method of Test for Diffuse-Light Transmission Factor of Reinforced Plastics Panels (D 1494-57 T) (Approved Dec. 13, 1957)

New Tentative.—This method, developed by Committee D-20 in cooperation with the Society of the Plastics Industry, provides a basis for characterizing reinforced plastics panels according to light transmission properties which are important in commercial applications.

ELECTRONIC MATERIALS

Tentative Specification for Nickel Alloy Cathode Sleeves for Electronic Devices (F 239-57 T) (Approved Dec. 13, 1957)

Revision.—The continuing interest of the electronics industry in standard test methods for the measurement and determination of properties of electronic tube cathodes has resulted in new methods of determination of physical, chemical, and metallurgical properties of these materials. This revision incorporates testing of shaped sleeves in the specification.

Actions on Standards

The Administrative Committee on Standards is empowered to pass on proposed new tentatives and revisions of existing tentatives and standards offered between Annual Meetings of the Society. On the dates indicated below the Standards Committee took these actions:

CEMENT

Tentative Method of Test for Calcium Sulfate in Hydrated Portland Cement Mortar (C 265-56 T) (Approved Dec. 13, 1957)

Revision.—Committee C-1 has revised this method to provide closer control of storage temperatures and a better indication of the rate and completeness with which gypsum reacts with the C_3A of the cement.

Tentative Method of Test for Flexural Strength of Hydraulic Cement Mortars (C 348-54 T) (Approved Dec. 13, 1957)

Tentative Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using Portions of Prisms Broken in Flexure) (C 349-54 T) (Approved Dec. 13, 1957)

Revision.—Changes incorporated in these two methods are designed to promote uniformity in specifications sponsored by various standardizing groups in the United States and Western Europe.

PAINT, VARNISH, AND LACQUER

Tentative Method of Test for Color Difference Using the Color Eye (D 1495-57 T) (Approved Dec. 13, 1957)

New Tentative.—Measurements of color and color differences are a fundamental requirement of the modern coatings industries. Several commercial instruments are now available and methods of test utilizing them have been prepared or are now in prepa-

NEW ASTM PUBLICATIONS

The ASTM publications described in these columns have just come off press, and may be obtained from Society Headquarters, 1916 Race St., Philadelphia, Pa.

Symposium on Railroad Materials and Lubricating Oils

The shift to diesel motive power in American railroads has brought about an urgent need for information on materials necessary for locomotives, their construction, operation, and maintenance.

This publication which developed out of two symposia held at the ASTM Second Pacific Area National Meeting in September, 1957 contains 17 papers on diesel fuels, lubricating oils, cleaning compounds, and nondestructive test methods.

Titles and authors of the papers are as follows:

RAILROAD MATERIALS

Introduction—Paul V. Garin
Operation of Diesel Locomotive with Dual-Fuel Systems—Paul V. Garin
Diesel Locomotive Lubricating Oil Requirements as Related to Fuels—J. L. Broughton and C. C. Moore
Performance of Residual Fuels in High-Speed Diesel Engines—D. R. Jones, K. L. Kipp, and J. E. Goodrich
Laboratory Research on Burning No. 6 Residual Fuel in an 8½ by 10 Opposed-Piston Diesel Engine—R. H. Beadle
Railroad Cleaners and Cleaning Procedures—J. L. Ramsey

Symposium on In-Place Shear Testing of Soil by the Vane Method

The purpose of this symposium, which was held at the ASTM 1956 Annual Meeting, was to acquaint the profession with a rather new tool for obtaining the in-place shear strength of medium to soft clay soils and organic silts. The Scandinavian countries have pioneered the development of the vane test and now use it widely in virtually all exploratory work where shear strengths are needed. There has been no attempt to standardize the test, although as this symposium showed much has already been accepted as standard. An additional purpose of the symposium was to bring in focus the developments made to date, so that the possibility of standardization could be considered. If it is possible to standardize certain elements of the vane test without stifling or restricting further progress through research and experimentation, then it would be desirable to do so.

The vane, which consists of four thin rectangular blades or wings brazed or

Standardization of Railroad Cleaning Materials and Methods for Diesel Locomotives—C. F. Jursch
Nondestructive Testing Applied to Railroad Materials and Equipment—A. S. Pedrick

LUBRICATING OILS

Introduction—C. M. Gambrill
Direct-Reading Spectrographic Control of Lubricating Oil Additive Manufacture—W. A. Rappold and R. E. Ramsay
The Application of X-Ray Spectrography to Refinery Control of Additive Metals in Lubricating Oils—E. N. Davis
Sampling of Lubricating Oil from Diesel Locomotives—W. K. Simpson
Methods for Spectrographic Analysis of Lubricating Oils—F. R. Bryan
The Evaluation of a Direct Emission Spectrographic Method for the Analysis of Used Lubricating Oils—W. D. Perkins, J. R. Miller, and J. H. Moser
Direct-Reading Spectrographic Evaluation of Used Railroad Oils—V. C. Barth
Chromatography of Lubricating Oils for Diesel Locomotives—E. R. Thomas
Filtration of Diesel Engine Lubricating Oil—S. L. Earle

STP 214. 176 pages. Price: \$4.50; to members, \$3.60.

welded to a small circular shaft, making a cross in section, is pushed into the soil and then twisted until the soil is ruptured. A cylindrical piece of soil very nearly the height of the vane and of the same diameter is thereby ruptured. From the maximum moment needed to rupture the soil and the surface area of the cylinder, the shear strength at rupture is easily computed.

Titles and authors of the papers are as follows:

Introduction—J. O. Osterberg
An Apparatus and Method of Vane Shear Testing of Soils—Harold J. Gibbs
Deep Vane Tests in Gulf of Mexico—Carl W. Fenske
Vane In-Place Soil Shear Device Developed and Applied by Oregon State Highway Department—W. C. Hill
The Use of a Field Vane Apparatus in Sensitive Clay—W. J. Eden and J. J. Hamilton

STP 193. Price: \$2.50; to members, \$2.

Symposium on Nondestructive Testing

The increasing importance of nondestructive testing as a method of materials inspection in all fields of engineering was exemplified at the ASTM Second Pacific Area National Meeting during which three sessions were devoted to a symposium on the subject. The papers from these sessions explore the latest and most widely used techniques and their applications in a variety of fields.

In addition to the symposium, a paper which covers the testing of bonded metals sandwich materials, from the Plastics Session of the same meeting, has been included in this publication.

Titles and authors of the papers are as follows:

The Activities of ASTM Committee E-7 on Nondestructive Testing
Committee E-7—Its Scope and Organization—J. H. Bly
Recommended Radiographic Practice—E. A. Burrill
Reference Radiographs—N. A. Kahn
Activities of Subcommittee III on Magnetic Particle and Penetrant Testing—Hamilton Migel
The Activities of Subcommittee V on Radiographic Procedure—C. H. Hastings
Report on the Activities and Future Projects of Subcommittee VI on Ultrasonic Testing—J. C. Smack
Summary—G. H. Tenney
Nondestructive Testing Applied to Railroad Materials and Equipment—A. S. Pedrick
Nondestructive Testing of Uranium—G. H. Tenney
Development of Techniques for the Detection of Cracks in Small Cylindrical Specimens by Reluctance Methods—Eugene Roffman
Electronic X-Ray Image Systems—R. F. Holste, J. E. Jacobs, and A. Pace
Fatigue and Ultrasonic Attenuation—Rohn Truell and Akira Hikata
Ultrasonic Testing of Aircraft Forgings—Alex Barath
An Ultrasonic Technique for Nondestructive Evaluation of Metal-to-Metal Adhesive Bonds—J. S. Arnold
Nondestructive Testing of Bonded Metal Sandwich Materials—R. E. Anderson

STP 213. 104 pages. Price: \$2.75; to members, \$2.20.

Symposium on Nondestructive Tests in Nuclear Energy Field Nears Printing Stage

By the time this BULLETIN reaches its readers the Symposium on Nondestructive Tests in the Nuclear Energy Field should be almost complete and in shape for final printing. The symposium was

held in Chicago, in April, 1957, with the American Institute of Chemical Engineers, Nuclear Division; American Nuclear Society; Society for Nondestructive Testing; Atomic Industrial Forum; and American Society for Testing Materials as joint sponsors. A large symposium with so many authors created a major publication task. Manuscripts for the most part have now been made available and have been cleared for publication with a considerable portion already in type.

Much of the information in the papers comprising the symposium was declassified just previous to April, 1957, and should prove of interest to industry in general.

Papers on Metals

This book contains eleven papers covering a wide range of topics associated with the properties of metals and their evaluation.

The increasing need for authoritative technical data in this field, spurred by aircraft and missile research, makes this a timely and worthwhile collection of papers. Practical experience and sound judgment based on long research are brought to bear upon modern concepts of corrosion, fatigue, and strength properties of metals.

The papers were presented at the 1956 Second Pacific Area National Meeting in Los Angeles. Titles and authors of the papers are as follows:

- Studies of Stainless Steel Columns Subject to Compression Loads—Julien Dubuc, George Welter, and V. N. Krivobok
- Shotpeening Effects and Specifications—H. O. Fuchs
- The Effect of Forming on Mechanical Properties—J. L. Waisman
- Axial Stress Fatigue, Creep, and Rupture Properties of Unnotched Specimens of Heat-Resistant Alloys—F. H. Vitovec and B. J. Lazan
- Determination of Young's Modulus Under Conditions of Relaxation—R. W. Fenn
- Effect of a Number of Variables on the Fatigue Properties of High Strength Steels—G. Sachs, B. B. Muvdi, and E. D. Klier
- The Effect of Temperature Frequency and Grain Size on the Fatigue of High Purity Aluminum—N. H. G. Daniels and J. E. Dorn
- Determination of Fatigue-Crack Initiation and Propagation in a Magnesium Alloy—Robert B. Clapper and J. A. Watz
- Uni-Directional Axial Tension Fatigue Tests of Beryllium Copper and Several Precipitation Hardening Corrosion Resistant Steels—M. H. Weisman, J. Melill, and T. Matsuda
- The Properties of Beryllium Copper Strip

as Affected by Cold Rolling and Heat Treatment—J. T. Richards and Ellsworth M. Smith

Pit Depth Measurements as a Means of Evaluating the Corrosion Resistance of Aluminum in Sea Water—T. J. Summerson, M. J. Pryor, D. S. Keir, and R. J. Hogan

STP 196. 182 pages. Price: \$4.50; to members, \$3.60.

Manual of Standards on Refractory Materials

This Manual includes all 41 ASTM specifications, classifications, methods of testing, definitions, and suggested procedures relating to refractories. It includes revisions to the Society's standards which were published in the previous edition in 1952 plus many additions, and is about 30 per cent larger than the 1952 edition. In addition to the ASTM standards, there is a proposed glossary of terms relating to refractories, their manufacture and use, and lists of standard samples of refractory materials which may be obtained from the National Bureau of Standards. Included also are surveys of refractory service conditions relating to the production of steel, cast iron, coke, copper, lead, portland cement, lime, and glass. There are both subject and author indexes.

414 pages; Price: \$5.50; to members, \$4.25.

Correction for 1957 Year Book

SEVERAL cross references to company memberships were omitted from the 1957 Year Book. Will you please have your book corrected by inserting the following in the list of members:

- CAMPBELL, HARRY A. (see Bell Aircraft Corp.)
- CAMPBELL, JOHN M. (see General Motors Corp.)
- CAMPBELL, W. E. (see American Society Lubricating Engineers)
- DAVIS, G. D. (see Nopco Chemical Co.)
- DUCHE, E. E. (see California Portland Cement Co.)
- KOENIG, JOHN H. (see Rutgers University, School of Ceramics)
- KREFFELD, W. J. (see Columbia University, Civil Engineering Research Laboratories)
- STOREY, E. B. (see Polymer Corporation Ltd.)

The cross reference GARIN, PAUL (see Southern Pacific Co.) has unfortunately, been transposed to an incorrect alphabetical section. It should appear on page 108 of the 1957 Year Book rather than page 107.

Errata in Methods E 87 - 56 T

The following corrections of errors should be made in Section 11 of the Photometric Methods for Chemical Analysis of Lead, Tin, Antimony, and Their Alloys (ASTM Designation: E 87 - 56 T):

In line 2 of Paragraph (d), delete "(NH₄)₂MoO₄." In line 3, change "0.53 g." to read "0.5 g."

In line 1 of Paragraph (e), change "5 g." to read "1 g." In line 2, change "0.5 g." to read "0.1 g."

ARE YOU NEW IN ASTM?



If you have only recently joined the Society or become a member of an ASTM committee, perhaps you may have wondered how the Society is organized, how technical committees operate and why they operate as they do. If so, you will be interested in a new booklet describing the how and why of technical committee operations to be available soon. The illustrated 20-page booklet entitled *So You Are Now a Committee Member* will be available free on request to members of the Society. Ask for the committee member booklet, addressing your request to ASTM Headquarters, 1916 Race St., Philadelphia, Pa.

Committee members who are not members of the Society, need not request the booklet because it will be mailed to them from Headquarters.



JANUARY 1958

NO. 227

NINETEEN-SIXTEEN
RACE STREET
PHILADELPHIA 3, PENNA.

Changes in ASTM Headquarters' Area

NUMEROUS changes have been taking place in the past two years in the area around ASTM Headquarters in Philadelphia. The most notable, of course, is the removal of the so-called "Chinese Wall" the large viaduct that carried the tracks of the Pennsylvania Railroad overground and over many of the city's north and south streets into the old Broad Street Station Building. A wide boulevard now occupies that space and new office buildings, a large new Sheraton hotel, and other buildings have been erected.

Nearer our own two buildings which back up to each other at 1916 Race St. and 1922 Cherry St. are several new smaller buildings. The Headquarters of the *Catholic Standard and Times* is a very attractive glass-fronted building and at the corner of 19th and Cherry the Allied Printing Industries of Philadelphia have a small modern building. All this of course is an improvement but more is to come.

Visitors to Headquarters now will hardly know the area because the six-, three- and four-story old red brick buildings immediately to the west of our Race Street building have all been demolished, and work starts soon on the erection of a new two and one-half million dollar auditorium and school for the Moore Institute of Art, one of our country's leading art schools for girls. The new dormitory, housing some 200 students, lies immediately to the west of our Cherry Street property and with the completion of the Institute's new building the complexion of the city block owned by the three educational institutions, the Academy of Natural Sciences, the Moore Institute of Art and ASTM, will have been changed markedly for the good.

Headquarters Expansion Study

Meanwhile, a special Committee on Headquarters Building Expansion,

headed by E. J. Albert, has begun its studies of the location of our Headquarters and the type of expanded buildings needed for the future ASTM Staff. This committee is part of a Long Range Planning Study Group now getting under way. The Society owns sufficient land adjacent to its Cherry Street property and in between the two buildings to erect multistory buildings which could house ASTM activities for many years to come. There is still some office space available, but within two years additional building may be required. Our

greatly increasing number of publications, which require additional storage, shipping facilities, and work area, and an expanded Staff, require constantly increasing space.

There are many who believe that the location of Headquarters close to downtown Philadelphia and along the beautiful Benjamin Franklin Parkway is excellent. This question and others will be studied by Mr. Albert's group which includes leading ASTM members and others who can help in reaching a consensus on the problems we face.

Members and Visitors Welcome

Meanwhile, as always, the latch string is out at Headquarters for our members and friends. We have many visitors including foreign delegations. Hundreds of leading technical men, engineers, and scientists, from overseas have stopped in to see us either on formally planned visits or informally, and the officers and Staff welcome these contacts. One never knows when walking into the ASTM lobby whether the visitor perusing our books or discussing some problem is from South Africa, the Philippines, or a couple of blocks away. All, however, are made welcome and usually find something of service and help.

R. J. P.

25-Year ASTM Members 1933-1958

In keeping with the desire of the Board of Directors to recognize longtime membership in the Society, the names of those who have completed 25 years this year are recorded below.

Ambelang, Carl E.
Anaconda Wire and Cable Co.
Austin, H. E.
Bigelow, Maurice H.
Birch, Raymond E.
Born, Sidney
Brown, Joseph A.
Chain Belt Co.
Coonley, John C.
Director, Government Test House
Falck, Giovanni
Felters Co., Inc., The
Filippi, Hugo
Gardiner Metal Co.
General Portland Cement Co.
Grede, William J.
Hecht, Max
Illinois, University of, Library
Imperial Chemical Industries, Ltd., Metals Division
Industrial By-Products and Research Co.
Interlaken Mills
Lauer, C. E.
LeFeber, Alfred
McMahon, Leonard H.

Metropolitan Water District of Southern California, The
Michigan State University Library
Miller, Durando
National Electrical Manufacturers Assn.
National Lubricating Grease Inst.
New York, State University of, College of Ceramics at Alfred University
New Zealand Public Relations
Newsome, Charles Todd
Phillips, Garnet P.
Phoenix Chemical Laboratory, Inc.
Secretaria de Recursos Hidraulicos Ingenieria Experimental
Stern, Cecil M.
Stivers, Earl R.
Swartz, Carl E.
Tennent, George R.
Timken Roller Bearing Co., Steel and Tube Division
Titley, Ralph H.
Waugh Equipment Co.
Waukesha Motor Co.
Western Felt Works

District Activities

President Kropf presents one of the 30 student awards at the Philadelphia District meeting. At the microphone is District Chairman Tinius Olsen II.

PHILADELPHIA

President Kropf Makes Presentation to 30 Students

The Philadelphia District, at ceremonies held at the Philadelphia Textile Inst. on November 25, awarded 30 Student Membership Awards to outstanding students from eleven colleges and universities in the District. Making the awards on behalf of the Society was President Richard T. Kropf. Tinius Olsen II, introduced the awardees.

Professor Percival Theel of the Textile Institute presided as toastmaster at the dinner preceding the ceremonies. Greetings were extended by B. W. Hayward, president of the Philadelphia Textile Institute and were acknowledged by R. J. Painter, Executive Secretary of ASTM. As guests of the Society, thirty members of a European Productivity Agency Mission currently touring the country attended the meeting. Jean Roger Barbaud from Paris, France, presented greetings on behalf of the delegation.

Following the presentation of the awards, the President addressed the meeting on "Extrapolation—2000 A.D." Based on his experience in longrange planning, Mr. Kropf explored the future of technology in the year 2000 A.D. He contended that while science would be able to provide food for an estimated seven to eight billion people in the world and enough materials and power to sustain a high level of industrialization, the critical shortage would come in scientific manpower. At the present rate of growth the United States alone would be short by 60 per cent of its needs for engineers and scientists. He urged that we stimulate youngsters to enter the physical science professions and that we make greater use of women scientists. He noted that almost 40 per cent of the Soviet scientists being graduated today are women.

NORTHERN CALIFORNIA

Marburg Lecturer Talks About Water

Everett Partridge, 1957 ASTM Marburg Lecturer, speaking on November 15 before a joint session of the National



Association of Power Engineers and the Northern California District of ASTM in San Francisco presented a picture of the problems associated with water.

Dr. Partridge reviewed the sources of our water supply, the quantities in-

involved and the uses to which it is presently put. He stressed the economic utilization of this natural resource, particularly in the industrial field where water under proper treatment can be and is reused a number of times.

Schedule of ASTM Meetings

This gives the latest information available at ASTM Headquarters. Direct mail notices of all district and committee meetings customarily distributed by the officers of the respective groups should be the final source of information on dates and location of meetings. This schedule does not attempt to list all meetings of smaller sections and subgroups.

Date	Group	Place
February 2-7	Committee D-2 on Petroleum Products and Lubricants	Houston, Tex. (Rice Hotel)
February 3	Southwest District	Houston, Tex.
February 10-14	Committee Week	St. Louis, Mo. (Hotel Statler)
February 18	Philadelphia District	Philadelphia, Pa. (Franklin Institute)
February 20-21	Committee D-6 on Paper and Paper Products	New York, N. Y. (Hotel Commodore)
February 20-21	Committee B-9 on Metal Powders and Metal Powder Products	New York, N. Y. (Sheraton-McAlpin)
February 24-26	Committee D-20 on Plastics	Pittsburgh, Pa. (Penn Sheraton Hotel)
February 25	Committee B-4 on Metallic Materials for Electrical Heating, Electrical Resistance and Electrical Contacts	Washington, D. C. (Mayflower Hotel)
February 26-27	Committee F-1 on Materials for Electron Tubes and Semiconductor Devices	Washington, D. C. (Mayflower Hotel)
February 26-28	Committee D-9 on Electrical Insulating Materials	Pittsburgh, Pa. (Penn Sheraton Hotel)
March 3	Committee E-13 on Absorption Spectroscopy	Pittsburgh, Pa. (Penn Sheraton Hotel)
March 5-7	Committee D-1 on Paint, Varnish, Lacquer and Related Products	Louisville, Ky. (Kentucky Hotel)
March 5-7	Committee C-16 on Thermal Insulating Materials	Hollywood, Fla.
March 18-21	Committee D-13 on Textile Materials	Washington, D. C. (Sheraton Park Hotel)
March 19	Chicago District	Chicago, Ill.
March 21	Washington, D. C. District	Washington, D. C.
March 25	Committee E-12 on Appearance	Washington, D. C. (National Bureau Bldg.)
March 26-28	Committee D-15 on Engine Antifreezes	Washington, D. C. (Shoreham Hotel)
April 1	Committee C-3 on Chemical Resistant Mortars	Headquarters, Philadelphia, Pa.
April 10-11	Committee C-23 on Sorptive Mineral Materials	Detroit, Mich. (Hotel Statler)
May 26-27	Committee D-10 on Shipping Containers	New York, N. Y. (Statler Hotel)
June 2-6	Committee E-14 on Mass Spectrometry	New Orleans, La. (Hotel Jung)
June 22-28	Annual Meeting	Boston Mass. (Statler and Sheraton Plaza Hotels)

"Diversity" Keynotes Annual Meeting

Broad Range of Materials and Methods to Be Presented in Technical Program in Boston

TYPICAL ASTM diversity will be displayed in the technical program shaping up for the Annual Meeting week of June 22 in Boston, Mass.

The "something for everyone" that is being planned by way of social, entertainment, and other features by the energetic Boston Committee on General Arrangements will be matched in the 12 symposia and numerous other sessions.

Following are the titles of the symposia and sessions which will be held under the sponsorship of ASTM technical committees. The April issue of the ASTM BULLETIN will carry the complete detailed program of the meeting.

SYMPOSIA

Effect of Water on Bituminous Paving Mixtures

Sponsored by Committee D-4 on Road and Paving Materials

Paper and Paper Products—New Developments with Accompanying Requirements for New Testing Methods

Sponsored by Committee D-6 on Paper and Paper Products

Radioactivity in Industrial Water and Industrial Waste Water

Sponsored by Committee D-19 on Industrial Water

Application of Soil Testing in Highway Design and Construction

Sponsored by Committee D-18 on Soils for Engineering Purposes

Particle Size Measurement

Sponsored by Committee E-1 on Methods of Testing

Solvent Extraction in the Analysis of Metals

Sponsored by Committee E-3 on Chemical Analysis of Metals

Durability

Sponsored by Committee E-6 on Methods of Testing Building Constructions

Fundamentals of Fatigue

Sponsored by Committee E-9 on Fatigue

Radiation Effects on Materials

Sponsored jointly by the Atomic Industrial Forum and Committee E-10 on Radioisotopes and Radiation Effects

Bulk Sampling

Sponsored by Committee E-11 on Quality Control of Materials

Materials Research Frontiers

Sponsored jointly by the Administrative Committee on Research and the New England Council

SESSIONS

Concrete

Sponsored by Committee C-9 on Concrete and Concrete Aggregates

Road and Paving Materials

Sponsored by Committee D-4 on Road and Paving Materials

Soils

Sponsored by Committee D-18 on Soils for Engineering Purposes

Textiles

Sponsored by Committee D-13 on Textile Materials

Properties of Metals with Rapid Heating

Sponsored by Joint Committee on Effect of Temperature on the Properties of Metals

Headquarters was host in November to members of European Productivity Agency Mission 388 shown here at the time of their visit to one of the testing machinery manufacturing companies near Philadelphia.—Photograph, courtesy of Tinius Olsen Testing Machine Co.



ASTM Sponsors Session at Standardization Conference

The American Standards Assn. held its first conference on the Pacific Coast at San Francisco, Calif., Nov. 13-15, 1957. The Eighth National Conference on Standards was held in conjunction with the 39th Annual Meeting of the ASA. Eleven sessions dealt with the development and use of standards for government and industry.

A panel discussion on that topic, a session on "Standardization in Your Company," and on "Cost Improvement Through Standardization," as well as "Partnerships in Standardization: Purchasing, Engineering, and Design," were followed by sessions which dealt with the application of standards to specific industries.

The session on Cost Improvement Through Standardization was sponsored by the ASTM. It comprised the following four papers:

Purchasing's Contribution to Cost Reduction Through Standards—H. W. Christensen, United States Steel Corp.

Employing Standards for Increased Productivity—Stanley R. Jepson, Eitel-McCullough, Inc.

Standardization from an Airline Viewpoint—R. D. Kelly, United Air Lines, Inc.

Prestressed and Precast Concrete—R. E. Tobin, Portland Cement Assn.

During the three-day conference, the Standards Medal of the ASA was awarded to ASTM Past-President John R. Townsend, Special Assistant, Office of Assistant Secretary of Defense, Research and Engineering.

Technical Committee Notes

First Meetings in Mexico for Cement and Concrete Committees

A COMBINATION of Mexican hospitality, sunshine, and semi-tropical surroundings provided the setting for one of the most successful meetings of Committee C-1 on Cement and Committee C-9 on Concrete Aggregates in recent years. Even though this occasion was not the first in which these committees have held meetings outside of the United States, the first being in Canada in 1955, it indicated to the committee officers that the geographical location did not affect the attendance of the members. Approximately 225 members of the two committees and their wives, some 90 in number, were in attendance during the five-day period at the Hotel Ruiz Galindo, Fortin de las Flores, Veracruz, Mexico. Local arrangements were handled by Alton J. Blank, Cementos Atoyac and Cementos Veracruz, a longstanding member of Committee C-1.

Despite the fascination of the surroundings, very serious technical committee work occupied the members most of the time, as evidenced by the reports submitted and actions taken.

Following two days of the committee meetings of Committee C-1 and preceding the meetings of Committee C-9, both groups enjoyed an inspection tour of nearby power plant installations and cement mills, and a typical Mexican barbecue. On this occasion, the ranking officials of the local cement interests were presented, preceded by a word of welcome from Julio Lacaud, President of Cementos Atoyac and Cementos Veracruz.

Cement Group Considers Concrete Testing by Cement Reference Laboratory

The extension of inspection services of the Cement Reference Laboratory to include concrete testing facilities was reviewed by Committee C-1 on Cement at its meeting on December 3. Four recommendations previously accepted by the committee and approved by the ASTM Board of Directors and the National Bureau of Standards have now been considered in detail. One of



Under a welcoming arch of gardenias members of ASTM Committees C-1 and C-9 and their wives gather for the meetings in Veracruz.

the items agreed upon was the fee to be charged for inspection services. This will be nominal and will not represent the entire cost of the inspection. It was felt very desirable to issue evidence of inspection in the form of a certificate which in no wise should be construed as certification of the work of the laboratory on a continuing basis.

Revisions were accepted which will incorporate in the Specification for Masonry Cement (C 91) a description of a tapping stick similar to that found in the Method of Test for Air Content of Hydraulic Cement Mortar (C 185). The revision also includes a change in the procedure for determining air entrainment. The Sponsoring Committee on Portland Cement discussed the need for closer collaboration between the Federal, AASHTO, and ASTM groups interested in specifications. A small change in the Specifications for Portland Cement (C 150) and for Air-Entraining Portland Cement (C 175) was considered, which will be in the

form of a footnote referring to the use of high-alkali cement.

The Working Committee on SO_2 Content reported on a cooperative study by five laboratories in which four cements have been tested for effect of SO_2 content using both cubes and bars. It has been found that the precision of the results of expansion and shrinkage tests is better than in the strength tests. However, the one-day strength test will be developed further and the effect of temperature will be studied using increasing increments of gypsum.

Round-robin tests nearing completion by the Working Committee on Strength included a series of larger-size prisms (40 mm).

A final draft of a method of test for fineness as measured by the No. 325 sieve is expected at the next meeting of the Working Committee on Fineness.

The measurement of plasticity of cement—a research problem—is being reopened by the Working Committee on Bleeding.

Three portland blast-furnace slag cements are being tested by the Working Committee on Heat of Hydration to determine the suitability of the present method of test (C 186) for use with this type of cement. Performance tests which will be reported in a paper are under way to establish data on sulfate resistance of portland blast-furnace and portland pozzolan cements. Revisions were approved in the sections on preparation of molding specimens and calculation of results in the autoclave expansion method (C 151).

One of the special features of the main committee meeting was the presentation of a paper on the Comparison of Cement Strengths in Mortars by Myron A. Swayze, Lone Star Cement Corp., Chairman of the Working Committee on Strength. Reviewing an extensive program of tests on 70 types of portland cement made at fifteen domestic and six foreign plants, the author concluded that the committee should consider replacing the briquet tensile strength with the compressive strength test in order to provide a single standard specimen yielding more concordant results.

Frederico Sánchez Fogarty, president of Camara del Cementos, speaking on the Mexican cement industry stated that 19 plants are now operating and taking care of the country's needs.

Work in Insulating Concrete Begun in Concrete Committee

The increasing use of lightweight and cellular-type concrete was recognized by Committee C-9, in the setting up of a new group on insulating concrete within the Subcommittee on Strength. This group which met during the December committee meeting, will consider the special problems which arise in connection with the testing of this type of concrete. It is recognized that the standard methods for determining properties of concrete do not necessarily apply to insulating. There has been considerable discussion on the adequacy of sulfur compounds for capping strength-test cylinders. A study of the effect of storage showed that there is no harmful effect on strength up to 28 days of storage. Further study will ascertain variations and further refinement of test procedures.

The size of concrete cores for testing referred to in the Method of Test for Compressive Strength of Molded Concrete Cylinders (C 39) was considered in view of some ambiguity in interpretation of the statements found in the method. It was agreed that a minimum diameter of 4 in. was desirable with the aggregate size not necessarily being of importance when the core was used for thick-



The beautiful setting of the committee meetings is glimpsed behind the officers of the committees, from left to right: W. S. Weaver, Secretary of Committee C-1; R. R. Litherer, Chairman of Committee C-1; W. H. Price, Chairman, Committee C-9; and Bryant Mather, Secretary of Committee C-9.



Committee C-1 at work. Front row, left to right: Federico Sánchez Fogarty, president, Camara del Cementos; L. C. Gilbert, ASTM Staff; and Stanton Walker, National Ready Mixed Concrete Assn.; Second row, third from left is E. J. Wechter, Louisville Cement Co., Chairman of Subcommittee on Masonry Cement; and fourth from left, W. C. Hanna, California Portland Cement Co., Chairman of Subcommittee on Methods of Chemical Analysis.

ness measurements only. For strength determinations it was agreed that the core diameter ratio of three times the maximum size of aggregate should apply. The use of vibration in the placing of concrete was also recognized as presenting a problem in connection with the preparation of test cylinders using the standard procedure of rodding of concrete. A suggested method now in use by some of the members, in which a

vibration procedure is incorporated, will be given study.

The means of determining the alkali reaction of aggregates in portland-pozzolan concrete will be included in a proposed test method which will be presented to the committee at its next meeting. Proposed test methods are now being reviewed covering the measurement of static modulus of elasticity

and Poisson's ratio in compression of molded concrete cylinders—applicable to diamond core drill specimens—and a method for determining creep of concrete in compression.

The Subcommittee on Pore Structure is now preparing a recommended practice on air content and air void characteristics including a cooperative research program on the relationship of pores and void characteristics. Approved revisions in the Standard Method of Test for Measuring Mortar-Making Properties for Fine Aggregate (C 87) include a broadening of the scope which now indicates that the method is only intended for the determination of the effect on mortar strength of organic impurities revealed by the colorimetric test. The tolerance of ± 2 per cent allowable in the weight and grading of test samples in the Los Angeles Abrasion Test (C 131) was extended to include grading A. Further study is to be given to a proposal that the coarse grade sizes E, F, and G in this standard be deleted.

The elimination of absorption requirements in the Specification for Lightweight Aggregates for Structural Concrete (C 330) as part of the basis for acceptance is being voted upon by subcommittee for possible recommendation at the next meeting. A proposed specification for cotton mats for curing—the same as specification M 73 of the American Association of State Highway Officials—will be recommended to the main committee. An extensive cooperative test program on fly ash, sponsored by the Subcommittee on Admixtures, now well under way, will lead to the development of a specification for fly ash as a pozzolan.

A number of detailed revisions were accepted in the Specification for Ready-Mixed Concrete (C 94) with a simultaneous recommendation for adoption as standard. The Method of Test for Bleeding of Concrete (C 232) was also approved for adoption as standard with editorial revisions. The Proposed Method of Test for Abrasion Resistance of Concrete as previously circulated was accepted for committee letter ballot. A second method for determining the abrasion resistance of concrete floors is now under study. Extensive revisions in the test methods referred to in the Specification for Packaged, Dry, Combined Materials for Mortar and Concrete (C 387) was accepted for letter ballot of the committee, thus correcting a condition which has not been found workable. An additional requirement of packaging and marking was approved by which all packages shall be identified as conforming with ASTM C 387 as well as to kind and type of material.

Soils Committee Meets Jointly with Mexican Group in Mexico City

Mexico City was a most appropriate site for the presentation of a group of papers on soils for engineering purposes, particularly foundations, prepared by authors from both Mexico and the United States. A joint meeting of Committee D-18 on Soils for Engineering Purposes and the Mexican Society of Soil Mechanics, an innovation in the history of the ASTM committee, proved to be a highly successful affair with an attendance of approximately 200 engineers and technical men. A series of five sessions, plus a meeting of Committee D-18, was held in the spacious and very modern auditorium of the School of Engineering, National University of Mexico, December 9-11.

The papers in session I on sampling were:

New Developments in Soil Sampling and Rock Coring Equipment—T. W. Van Zelst

Consolidation of Mexico City Volcanic Clay—L. Zeevaert

Laboratory Consolidation Tests for Expansive Clays—R. F. Dawson

Papers in sessions II and III, relating to the subject of compaction and compaction control, were:

Some Factors Affecting the Dynamic Compaction Test—E. Tamez

Compaction Characteristics of Gravelly Soils—W. G. Holtz and C. W. Lowitz

Effect of Tire Pressures and Lift Thicknesses on Compaction of Soil with Rubber-Tired Rollers—W. J. Turnbull and C. R. Foster

A Rapid Method of Construction Control for Embankments of Cohesive Soil—J. W. Hilf

Investigation of a Volcanic Soil for the Construction of an Earth Dam—E. Rios Lazcano

Sessions IV and V included:

Soil Deformations Under Repeated Stress Applications—H. B. Seed and R. L. McNeill

Unconfined Compression and Vane Shear Tests in Volcanic Lacustrine Clays—R. J. Marsal

Testing Soils with Transient Loads—R. V. Whitman

Session V, consisting of miscellaneous subjects, contained:

Seepage Determination on Hydraulic Structures by Means of Radioactive Isotopes—I. Saint Ortiz

Sinusoidal Surface Waves in Stratified Soils—J. J. Slade, Jr.

Underpinning and Straightening of an Eleven-Story Building in Mexico City—W. Streu

A very interesting as well as educational all-day tour of soil and founda-

tion conditions and projects within the Valley of Mexico, within and surrounding Mexico City, was held on the final day. A recent earthquake provided additional opportunities for observation of soil foundations.

Members of Committee D-18 and their wives expressed pleasure with the program arranged by the officers of the Mexican Society of Soil Mechanics, not only in the facilities for the technical sessions but also in the social program provided for both the ladies and the participants in the sessions.

The conference papers have been prepared in published form in both Spanish and English and it is expected that discussions will be added. Information on the availability of these papers can be obtained from ASTM Headquarters.

Halogenated Solvents Subject for New Committee

On Dec. 4, 1957, a conference was held at ASTM Headquarters representing industry interests in cleaning solvents both of the types used for cold-dipping and cleaning of electric motors, etc., and for vapor degreasing. Also present were representatives from several ASTM committees that have some interests in solvents. The conference reviewed the present ASTM work on solvents, including those for paint developed in Committee D-1 on Paint, Varnish, Lacquer, and Related Products, petroleum-based solvents in Committee D-2 on Petroleum Products and Lubricants, and aromatic solvents developed by Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials. The conference also considered the work in Committee D-12 on Soaps and Other Detergents which has a special interest in development of tests for cleaning efficiency. Committee D-12 also sponsors a bibliography on metal cleaning.

The conference reviewed the relation of the proposed work to that of ASA Sectional Committee 237 on Toxic Dusts and Gases.

It was evident from the discussions that standards are needed for the kind of solvents used for cleaning and, in particular, the halogenated organic solvents which are not covered by any existing ASTM committee. Accordingly, the conference recommended the establishment of a new committee covering halogenated organic solvents and solvent mixtures having halogenated solvents as a component.

The recommendation will receive the attention of the Board of Directors.

Non-Ferrous Metals

Titanium and Zirconium Standardization Under way

Recognizing that titanium products are now an established commercial item, Committee B-2 on Non-Ferrous Metals has formally organized a new Subcommittee IX on Titanium and Titanium Alloys with T. W. Lippert, general manager, Titanium Metals Corp. of America, as chairman. Formerly this work fell under Subcommittee VIII on Miscellaneous Refined Metals and Alloys of Committee B-2. There are approximately fifty members of the new subcommittee, balanced between producers and consumers of titanium metals.

The first meeting was held in Pittsburgh on December 5, with an excellent attendance in spite of inclement weather and a transportation strike in the city.

The sponge specification (B 299 - 55) is being revised in the light of advances in the industry. The most significant change is the addition of a 120 grade to accompany the present 140 and 160 grades. The lower Brinell hardness reflects less impurity content in this basic product.

The present Specification B 265 - 52 covering commercial titanium grades of wrought products will be replaced by several new specifications. The new specifications will include titanium alloys and several grades of commercial titanium. It is expected that specifications will be written covering (1) plate, sheet, and strip; (2) rod and bar; (3) tubing; (4) pipe; and (5) wire. Industry-wide size tolerances have now been established on all these products, as well as check tolerances for chemical analysis. The Chemical Industry Advisory Board of the American Standards Assn. has expressed a need for a specification for titanium pipe. Also the ASME Boiler and Pressure Vessel Committee is writing a special case for the use of titanium in pressure vessel construction. The new Subcommittee IX intends to cooperate with both these groups.

A great need was expressed for a simple codification system for titanium and titanium alloys. Several have been attempted but as yet none have been universally adopted. Mr. Du Mont, Titanium Metallurgical Lab., Battelle Memorial Inst., was appointed to draw up a simple memory system, along the lines of that developed for steels, for consideration as an ASTM document.

The next meeting of Subcommittee IX of Committee B-2 is scheduled for Thursday, February 13, 1958, in St. Louis. The meeting will be in conjunction with 1958 ASTM Committee Week lasting from February 10 to 14.

Zirconium Task Group Membership Open

A survey reported at the ASTM 1957 Annual Meeting, revealed a need for industry-wide specifications for zirconium. Committee B-2 has authorized its Subcommittee VIII to write such specifications, and a task group is being organized for this purpose. The Chairman is W. W. Stephens, The Carborundum Metals Co., P. O. Box 32, Akron, N. Y. Membership on this group is open to those who make proper application to Mr. Stephens. Anyone involved in the manufacture, fabrication, or use of zirconium metal should have a vital interest in this development.

Effect of Temperature on Metals

Survey of High-Temperature Properties of High-Chromium Steels Completed

AT THE meeting held in New York City, December 4, the Data and Publications Panel of the ASTM-ASME Joint Committee on Effect of Temperature on the Properties of Metals announced completion of a survey on the elevated temperature properties of high-chromium alloy steels. The data covering this survey will be published by ASTM as a special technical publication, completing a three-part project in which the first two phases on plain-carbon and medium-carbon alloys were published as ASTM STP 180 and STP 199, respectively.

In reviewing some of the recent accomplishments of the Joint Committee, G. V. Smith of Cornell University, chairman of the Data and Publications Panel, announced completion of a survey on the strength of weldments. The report presents tensile, rupture, and creep properties for carbon, low-alloy, and austenitic steel and for a number of complex alloys developed for high strength at high temperatures. This work will be published by ASTM as STP 226 under the title "The Elevated Temperature Properties of Weld-Deposited Metal and Weldments."

J. D. Lubahn of the General Electric Co., chairman of the General Research Panel of the Joint Committee described literature surveys recently completed on notch-rupture behavior and cyclic loading. As a following step, Lubahn stated that an extensive research program will be undertaken on notch-rupture behavior under particular applications.

The Test Methods Panel has under way a program for providing standard test specimens for calibrating creep and rupture test equipment. The use of calibrating specimens may obviate the necessity for defining the precision of temperature measurement and control in testing of this sort.

The Joint Committee has a continuing program of sponsoring papers and symposia based on their extensive research. It is largely through the cooperation and contributions of many organizations that this work is possible.

Symposium Correlates Functional Properties of Wax with Physical Properties

TECHNICAL COMMITTEE M on Petroleum Wax, a joint activity of the ASTM and the Technical Association of the Pulp and Paper Industry (TAPPI), will hold a Symposium on Wax on Feb. 19, 1958, in the Commodore Hotel, New York City, in conjunction with the annual meeting of TAPPI.

A full-day's program is planned by the wax group which functions as a subcommittee of Committee D-2 on Petroleum Products and Lubricants.

Titles and authors of the papers which will be presented are as follows:

- Sealing Strength of Wax-Polyethylene Blends—D. S. Brown, W. R. Turner, and A. C. Smith, Jr., The Atlantic Refining Co.
- The Relationship of Wax Crystal Structure to the Water Vapor Transmission Rate of Wax Films—R. C. Fox, California Research Corp.
- Composition Versus Properties of Microcrystalline Waxes—W. P. Ridenour, I. J. Spilners, and P. R. Templin, Gulf Oil Corp.
- Solid Petroleum Hydrocarbons and Their Effect on Wax Properties—R. T. Edwards, Socony Mobil Oil Co., Inc.
- Relation Between Composition and Blocking Temperature of Paraffin Waxes—K. G. Arabian, Shell Oil Co.
- Effect of Refining Conditions on Wax Quality—R. L. Murphey and W. B. Chandler, Jr., The Texas Company
- Gloss and Gloss Stability of Waxed Papers—F. J. Hughes and D. C. Walker, Standard Oil Co. (Ind.)
- The Effect of Paper-Making Materials on Wax Oxidation Rates—G. G. Rumberger, Marathon Corp.
- Factors Influencing the Staining Tendency of Wax—Joseph Phillips, L. Sonneborn Sons, Inc.

By W. H. LUTZ¹

TECHNICAL men, who are concerned with the manufacture of engineering materials, find themselves not infrequently embroiled in customer complaints. An ordinarily well-behaved process or product suddenly develops "bugs." An initial complaint occurs; it is followed by a few more complaints. The mild distress first registered by the technical man's sales organization soon changes to anguished cries for action, if the situation worsens. The technical man is now faced with a problem that has all the urgency of a three-alarm fire.

The answers to a hundred questions are needed at once. What went wrong? Is the problem generalized or is it confined to a few batches? How did these batches differ from satisfactory batches? How can future trouble be avoided?

¹ Vice-President, Pratt and Lambert, Inc.

² Copies of the collection of Unsolved Problems published in 1954 are available on request from ASTM Headquarters, 1916 Race St., Philadelphia 3, Pa.

Can anything be salvaged from the complaint materials?

The stage is now set for an unwanted research project—not a nice clean-cut leisurely search for basic knowledge, but a crash program aimed at developing answers to a very specific and very demanding problem. Unhappy customers are impatient customers. The fires of hell never burn hotter than the breath of a complaining customer.

Time passes. Exploratory investigations followed by sweat, laboratory work, and a lot of accelerated testing, usually reveal the source of trouble. The process or product is righted and emotional and business tensions ease.

This experience will have been a broadening one and it will have been character-building, if nothing else. Knowledge of materials—no matter how painfully gained—does increase the effectiveness of materials. Both men and materials are forged in the high pressures resulting from complaints on materials. Trouble provides the incentive to devise new tests and evaluate

old ones, to look over specification requirements, and to loosen or tighten specification limits. In other words, it is a hidden resource for evolving new information. Many an ASTM specification owes its existence or certain of its requirements to complaints about a product or process. Not all complaints yield to a quick attack and some attain the status of an industry-wide problem. In the latter case, they might justify inclusion in the ASTM list of "Some Unsolved Problems,"² which has recently been given the more appealing name "Challenges in Materials Research."

If you, or the technical committees to which you belong, know of any such problems, please submit them to the Administrative Committee on Research. Be sure to state the problem, give a concise review of present knowledge, list questions that need answering, and provide literature references, and the names and addresses of individuals from whom further information can be obtained.

RR Diesels CAN Use Residual Blend Fuels

Wherever diesel engines are used for motive power, economies are possible through the use of fuels requiring a minimum of refinement. This subject was covered in considerable detail in the Symposium on Railroad Materials sponsored by the Society at the West Coast National Meeting in 1956. This symposium is now being published and will be available soon. A description may be found on page 16. Included in the symposium publication is a comprehensive report by Paul V. Garin describing the research conducted by the Southern Pacific Co. which has resulted in the proved feasibility of a dual fuel system in which a so-called "economy" distillate fuel is used for idling and low speeds and a heavier blended fuel is used from about half to full power. Mr. Garin's paper originally included a rather extensive summary which does not appear in the symposium publication. This summary in somewhat abridged form is presented below since it would appear to be of interest to readers of the BULLETIN.

Since 1954 the Southern Pacific Co. has successfully used economy distillate fuels with minimum cetane rating of 34 and maximum sulfur content of 1 per cent in its diesel locomotives. This came about through an extensive research program to use less critical fuels. Continuation of the research since 1954 has resulted in the development of a dual fuel system for operation of locomotive diesels on distillate fuel when idling or at low outputs and on a less critical fuel, such as a residual blend, at high outputs.

Electric-motive diesel engines, as presently designed, require suitable distillate fuels for idling and low outputs to avoid fouling of engines, excessive smoking, and spark emission. Above about the middle throttle position, the engine investigated by the Southern Pacific Co. will operate effectively on residual blended fuel (referred to as X-6 type).

It has been found that engine performance, availability, and utilization are not adversely affected by dual fuel operation.

In order to use fuels like X-6 which have high viscosity (300 SUS at 100 F), it is necessary to preheat the fuel prior to injection. This is done with heat exchangers utilizing waste heat from engine cooling water. Still higher viscosity fuels could be heated, taking advantage of waste heat from the exhaust stack to supplement that from the cooling water. Electric fuel heaters are not economical because this energy is chargeable against the output of the diesel engine.

It has been found that the combustion pattern with X-6 fuel, when heated to 170 F, is essentially the same as with distillate fuel when operating at full throttle. Also, the amount of smoke emission of the X-6 fuel is comparable to that of distillate fuel indicating complete combustion. Spark emission is normal.

Although it might be expected that the high sulfur content of 3 per cent of the X-6 fuel would cause excessive wear or corrosion, this was not found to be

the case, no doubt because the fuel is used only at high output when engine temperatures and pressures are conducive to complete combustion.

The cetane number of 25 for the X-6 fuel does not appear to affect performance.

Filtration of the heavy blended fuel is accomplished with a sock type filter element as a primary filter and a resin impregnated paper type element as a secondary filter. No difficulty was experienced with this type filter.

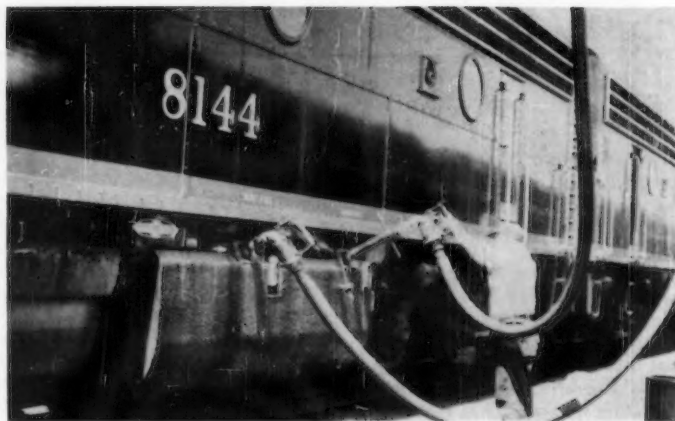
Of the various injectors investigated, a standard injector with spray tip having 0.115 in. diam holes with 75-deg spray angle has produced good results. Alternate operation of injectors on the two types of fuel seems beneficial in minimizing tip deposits.

The higher volumetric heat content of the X-6 fuel, even when heated for injection, increases the horsepower output of the engine at a given power piston setting. A secondary saving can, therefore, be effected by setting the power piston to compensate for the increased heat content in the lower gravity X-6 fuel.

Examination of the engine parts after a period of operation indicates that while the wear is greater than that experienced with distillate fuel only, it was much less than anticipated. While chromium-plated cylinder liners were found to hold up well in dual fuel service, it was evident that the type and surface finish of the chromium plating influences the wear rate of these parts. Further investigation of this is in progress. It is interesting to compare the wear rate determined for the X-6 fuel with that for the distillate fuel. Based on the same type of chromium-plated liner, the wear rate per year at a position 2 in. from the top of the liner was found to be 6.4 mils for X-6 fuel and 4 mils for the distillate fuel. The engine builders' recommended limit of wear for cylinder linings is 25 mils measured across the diameter.

The top compression ring wear with X-6 fuel was found to be about double that with the distillate. Oil-ring wear on the other hand was comparable for the two fuels. The wear on the piston thrust washers was found to be about 1.7 times more with the residual fuel. On the other hand, the wear on connecting rod bearings and journals as well as piston pins and bushings was about the same for both fuels.

Comparisons of engine deposits found after 13½ months of operation with X-6 fuel with that of an engine operating on the distillate fuel after 12 months of operation using the same lubricating oil, rated the X-6 unit as 61.9 per cent clean



Fueling of two-compartment tank on diesel dual fuel unit.

and the distillate fuel as 69.5 per cent clean, 100 per cent representing a completely clean engine.

Certain other deposits were investigated such as compression ring belt deposits, air port deposits, and those around the valve stems and, in general, they were found either comparable for the two fuels or somewhat higher for the X-6 fuel.

In order to minimize corrosive attack on valves and other parts in contact with hot exhaust gases, an investigation is being carried out to evaluate protective coatings such as aluminizing and chromium plating. Stellite seats on exhaust valves were found desirable to increase resistance to guttering caused by particles, airborne or otherwise.

As was expected, the lubricating oils from the X-6 test unit were found to contain somewhat higher combustion residue loads than with the distillate fuel. Therefore, it is necessary to drain the oil more often when using the X-6 fuel. Preliminary experience with non-dispersant lubricating oil in dual fuel service was promising. Analysis of the oil during the initial test period showed low acid numbers, stable viscosity, and low percentage of contaminants. This was probably due to the improved filtration possible with this type of oil permitting removal of contaminants through filter action. While lubricating oil practice continues to be investigated, and it is evident that further improvements are possible, the use of the dual fuel system has not required any major changes in normal lubricating oil practices. It is believed that improvements can be made, especially in the filter system, to remove contaminants more effectively.

In general, the performance of the dual fuel system as developed in the

program of the Southern Pacific has been very satisfactory both on the original test units and on a large number of locomotive units equipped with this system and employed in "fleet tests." The dual fuel equipment can be considered fully reliable and suitable for regular locomotive service. No insurmountable maintenance problems have been encountered although certain aspects require more study and improvement. This applies particularly to the improvement of the top compression ring and exhaust valves for the diesel engines. Until more satisfactory lubricating oils can be developed for this operation, more frequent oil drains will be required. Many disadvantages in the use of the dual fuel system are more than overcome by the increased economy resulting from the use of these fuels.

Viscometer Calibration Discontinued

IN LINE with the Federal Government's general policy of withdrawing from activities that can be carried on by private industry, the National Bureau of Standards will discontinue calibration of viscometers on January 1, 1958. This action will permit the Bureau to devote more of its resources toward meeting demands of science and industry for new and accurate standards.

Viscometers are widely used in industry to observe the flow characteristics of nonrigid materials. However, most users of these instruments now carry out the calibrations in their own laboratories using viscosity standards available from the NBS. Inquiries addressed to interested parties have brought forth no substantial objections to discontinuance of the service.

Random Samples...

FROM THE CURRENT MATERIALS NEWS

From the broad stream of current materials information flowing from "in-box" to "out-box" in a busy editorial office, random samples (mostly random) have been plucked. Thinking them worth re-showing to ASTM'ers who may have missed the original articles, we have included them here. Of course, we had to trim the samples to fit. There will be those who are not satisfied with samples, especially ones which are not really random. But these ASTM'ers can contact the institution, magazine, governmental agency, etc., who placed the original information in the stream, or address Random Samples, ASTM, 1916 Race St., Philadelphia 3, Pa.

Lion in a Cage

COLUMBIUM has moved a step closer to its expected use in rockets, missiles, jet engines, and nuclear reactors of the future.

Westinghouse scientists have developed a technique for the ultrapure preparation of this rare and little-known chemical element. Preparation of the metal in its purest possible form is essential to a basic understanding of the properties that make it of interest as a high-temperature structural metal. The high purity was produced by a metallurgical process discovered at the Westinghouse Research Laboratories and called "cage zone melting." It had produced the first columbium ever made on a scale large enough for realistic scientific study of the ultrapure metal. Their process removes all but the very last traces of impurities in columbium by refining the metal at a white-hot temperature of almost 4400 F while confining it under a vacuum only four billionths the pressure of the atmosphere. This is thought to be the first time that any metal has ever been kept in the molten state under such simultaneous extremes of high temperature and low pressure.

Columbium has unique high-temperature properties. Unlike most metals, it does not lose its room-temperature strength drastically with increasing temperature. At 2000 F—just about the melting point of copper—it has roughly the same strength as molybdenum and is several times stronger than the best available nickel-base and cobalt-base alloys at this temperature. Columbium also has valuable structural characteristics. It is only slightly heavier than iron and is not brittle at room temperatures as are other high-temperature metals such as chromium and molybdenum.

The preparation of high-purity columbium is a very important aspect of the research program on columbium. The ultrapure metal makes possible detailed studies of the effects produced by oxygen, nitrogen, and other elements which combine so readily with the metal. This understanding will aid the design of columbium-base alloys that will com-

bine the excellent high-temperature strength of the metal with improved chemical inertness.

Because it is so active chemically near its melting point of 4380 F, molten columbium must be handled in a vacuum or in an atmosphere of some inert gas such as helium or argon. For the same reason, columbium of extreme purity cannot be prepared by melting it in any sort of crucible or similar container.

Westinghouse "cage zone melting," invented at the Westinghouse Research Laboratories three years ago, solves both of these problems at the same time. A rectangular bar of impure columbium stands vertically on a movable platform, which can move the bar up and down inside a coil of heavy copper tubing. The coil and platform are enclosed in a sealed vessel having a vacuum only four billionths the pressure of the atmosphere. When a strong, high-frequency electrical current is sent through the coil, corresponding currents are induced in the bar, which melts from the inside out. The four corners of the bar do not melt, but form a "cage" within which the molten columbium is trapped. The bar thus serves as its own crucible within which the white-hot metal is refined. As the bar rises through the coil, a molten zone of columbium travels down the bar from top to bottom, melting the bar progressively throughout its entire length. Successive "passes" through the coil result in columbium of increasing purity as more and more impurities are removed.

New Adhesive Holds 5000 psi

RESEARCHERS at Tennessee Eastman Co., Kingsport, Tenn., a division of Eastman Kodak Co., have come up with an adhesive which is said to be unusually strong, versatile, and quick-setting.

A single drop on a surface 2 in. in diameter holds 200 lb in 5 min, 5000 lb in 30 min, 15,000 lb in 48 hr. It requires no heat, pressure, or solvents. (Ultimate steel-to-steel tensile strength: about 5000 psi in 48 hr.) Glass-to-glass bonds are unbreakable in 5 to 15 sec, steel-to-steel in 20 sec, wood-to-wood in 5 min.

Called Eastman 910 (acryanoacrylate), the adhesive bonds unlike materials which do not respond to conventional cements, and firmly holds small joints such as those in miniature assemblies. It works well on many plastics.

Bonding surfaces must be thoroughly cleaned and dried. Only surface dirt needs to be removed. A toluene-acetone mixture is recommended for metal surfaces. The adhesive is applied to one side of the joint. When a thin film covers the whole surface, the other half is pressed firmly in place.

Eastman says that 1 lb will cover 100 sq ft on a smooth, nonporous surface. The adhesive sets within a few seconds after the parts have been pressed together. Most bonds are sufficiently cured within an hour to withstand rough handling.

Short exposure to 212 F does not materially affect tensile strength; prolonged exposure breaks it down. Eastman 910 resists solvents well. A 24-hr immersion in alcohol, benzene, or acetone does not affect it. Weak alkalis or acids reduce tensile strength slightly.

It is pointed out that the adhesive reacts quickly with alkaline substances. It must be kept in closed containers. Shelf life is six months to one year. It is not harmful to skin although the dried film is hard to remove.

Steel, The Penton Publishing Co.

Evolution of the Waterhole

A CHEMICAL shield is being tested at the Rattlesnake Reservoir in Colorado to determine whether a practical way can be found to reduce evaporation from lakes and reservoirs and save vitally needed water in arid and semiarid regions.

Laboratory studies indicate that up to 65 per cent of evaporation losses can be eliminated with a surface coating of hexadecanol, a chemical which has no known harmful effects on human or aquatic life. Western reservoirs lose between three and eight feet of water each year through evaporation.

The hexadecanol being used is in flake form, resembling soap flakes.

(Continued on page 96)

THE TOWER OF BABEL

By ARTHUR W. CARPENTER

This address by Past President Carpenter was given on the occasion of his receiving the Charles Goodyear Medal of the Rubber Division of the American Chemical Society.



IN THE ruins of the city of Babylon, which were excavated by the Germans in the years before World War I, were exposed the remains of the Tower of Babel. This was the tower of the sanctuary of the great Babylonian god Marduk and was opposite the temple of Marduk on the Euphrates which was restored with great splendor by Nebuchadnezzar about 600 B.C. This structure, which had more than once been destroyed and rebuilt, was, at the time of the Babylonian empire, nearly 300 ft high. It was built in seven stages, one above the other and the length and breadth of the lowest stage was the same as the height. The only account of its original construction is that of the Bible in the Book of Genesis, Chapter 11, for this event occurred in the dim past, some time between the great flood of about 4000 B.C. and the days of the patriarch Abraham about 1900 B.C. To the descendants of Noah who built it, their whole earth was indeed of "one language and one speech." Living harmoniously together, they undertook to build a city and a tower "whose top may reach unto heaven." The Lord, when he saw the work, said, "Behold, the people is one and they have all one language; and *this* they begin to do: and now nothing will be restrained from them, which they have imagined to do." Whereupon, He confounded their language that they might not "understand one another's speech" and "scattered them abroad from thence upon the face of all the earth, and they left off to build the city. Therefore is the

name of it called Babel; because the Lord did there confound the language of all the earth." In this the name Babel is taken as having been derived from the Hebrew "balal," meaning confusion, but another and perhaps better derivation would be the Assyrian "babili" meaning "Gate of the God."

This story from the Bible came to mind when you gave me the great honor of being chosen as Goodyear Medalist of 1957 because the physical and mechanical testing of rubber has often exemplified the confusion which exists when people do not understand each other because of conflicting data. When, through research, development, and standardization, these people become one and are given the same language, there is, in truth, little which "will be restrained from them which they have imagined to do." Cooperative effort in attaining unification has had much to do with the progress which has been made in the 30 years I have worked in the field of rubber testing. This Goodyear Medal Award is therefore a recognition not only of my contribution but also of those of my associates in the B. F. Goodrich Co. and in the committees of the American Society for Testing Materials and the Division of Rubber Chemistry of the American Chemical Society.

When Charles Goodyear discovered the process of vulcanization in 1839, he had no physical means other than hand tests and visual observations to help him evaluate his product. The final measure of utility was obtained by trial in the intended service with the result that large losses were often sustained before conclusive answers were available. The unusual elastic behavior

of vulcanized rubber, however, early inspired studies of the relation between applied stresses and the resulting deformations. Compressive loads were used in the earliest work, probably because of employment of the material for railroad bumpers. Later, the investigations were greatly extended using tension and torsion. The work of the pioneers in this field, Hovine (1),¹ Debonnefoy (2), Boileau (3), Villari (4), Stévant (5), and Mallock (6), has been often referred to in the rubber literature, but it is interesting that Boileau in 1856 noted the difference in effect during loading and unloading now known as hysteresis and that Villari's data, published in 1869, for stress and strain to the breaking point show, when plotted, a typical stress-strain curve for rubber. These men were seriously handicapped by lack of knowledge about rubber and suitable facilities for their investigations. Only the simplest apparatus which the experimenter himself devised was available.

In tension testing, the rubber specimen was hung vertically from a hook and the stress was applied by weights placed in a loading pan attached to another hook at the lower end. Elongation was read between gage marks. The problem of gripping the rubber threads or strips was solved by bending the ends to form loops and binding them while under strain. Stévant was the first to use ring specimens. Today, the variation in load application by adding weights by hand could not be tolerated, but these early investigators did not regard it as seriously influencing the results. Except for these men and a few others who were gradually acquiring scientific knowledge of the

NOTE.—The boldface numbers in parentheses refer to the list of references appended to this paper.

fundamental physical characteristics of vulcanized rubber, testing of the material, as we know it, cannot be said to have existed prior to the dawn of the 20th century. It was a time when men concerned with rubber were busy in extending the benefits of Charles Goodyear's discovery, building factories, trying out and commercializing the wonderful material he made available in suggested uses, numbering more than 500, which he described in Volume II of his book entitled "Gum-Elastic" published in 1853.

During this period, chemical testing also languished, for it was a time when the rubber industry was in the hands of practical men whom Disraeli has defined as "those who practice the errors of their forefathers." Of course, chemical analysis had been used for scientific investigations of rubber ever since Michael Faraday (7) had determined the ratio of carbon and hydrogen in 1826. After Goodyear's discovery of the action of sulfur and heat and Parkes' discovery (8) of cold vulcanization by sulfur monochloride, both methods seem to have been used for nearly fifty years in the manufacture of rubber goods with little attention to the chemistry of the processes. Toward the close of the century however, C. O. Weber (9) published early results of his extensive studies of the chemical nature of vulcanization, which were followed in 1902 by his classic book on "The Chemistry of India Rubber" which summarized the chemistry and technology of rubber known at the time. The controversies following these publications attracted chemists to this new field and led to many additional investigations. One may suppose also that the interest of the rubber manufacturers was aroused because within a few years they began to install laboratories and to employ technically trained people. The start was slow as is evidenced by the statement of Oenslager (10) on the occasion of the Perkin Medal Award in 1933 that 25 years earlier he was one of three chemists engaged in the industry in Akron.

A consequence of the work of Weber and other investigators was to impress the rubber manufacturers with the im-

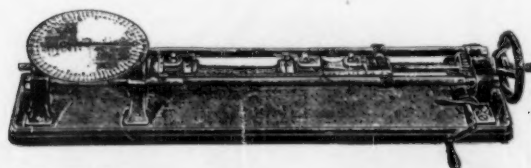
portance of chemical determinations of free and combined sulfur, and the laboratories of the industry began using these measurements to indicate the state of vulcanization. Also, the presence of large amounts of resins or added softeners was regarded as indicating a poor quality of product because high amounts of natural resins were characteristic of the lower grades of the wild rubbers of the period. Thus, great reliance came to be placed in chemical testing, and many of the early rubber chemists were definitely prejudiced against all tests of quality which were not based on chemical analysis. As late as 1911, J. G. Pol (11) stated that only chemical tests were made at the Dutch Government Bureau for India Rubber Trades and Industry in Delft.

During the first decade of the new century, the stage was rapidly being set for the tremendous changes which were ahead, including not only enormous expansion of production but also development of new concepts of scientific research and technical control of manufacturing through physical as well as chemical testing. Schidrowitz (12) has noted that Weber made no reference to the classic studies of Stévant on elastic properties of rubber and that he referred to testing for tensile properties in very rudimentary form, recommending that such tests be made in an ordinary cloth testing machine. A year after Weber's book, Bouasse and Carrière (13) published their work on cycles of extension and retraction using loads continuously applied by feeding copper chain into a container attached to the lower end of the specimen. This was followed by a superb treatise by Bouasse (14) on the elastic moduli of rubber. Soon thereafter, Pierre Breuil (15) began a series of articles which at the time must have been considered revolutionary, for he wrote about mechanical tests used for manufactured rubber and compared them with tests of metals. He discussed the effects of many variables and described methods and equipment then employed in the laboratories of the Conservatoire des Arts et Métiers. Testing machines, built on principles still widely used, were illustrated, including lever and pendulum types as

well as the small hand-operated Delaloe machine (Fig. 1) employing a calibrated spring. The motion was usually obtained by screws operated mechanically or by hand, and even hydraulic machines were included. Both ring and straight test specimens were used, and a dumbbell shape was shown. Some attention was also given to methods which would now be classed as specification and performance tests.

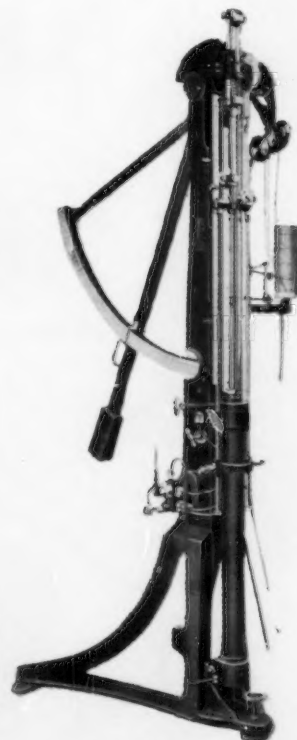
Physical Tests Spurred by NBS

In 1906, the year that Oenslager made the discovery of organic accelerators of vulcanization, an event occurred which was ultimately to have vast influence on the acceptance of physical testing. The U. S. National Bureau of Standards (16) became interested in rubber because of its desire to assist other Government agencies in the preparation of purchase specifications. The automobile industry was about to commence its tremendous development, and everywhere technical activity was rapidly growing. In 1907, though not reported until 1916, Dr. Geer (17) started his work at the B. F. Goodrich Co. laboratory on the problem of age deterioration of rubber. The following year, at a conference in connection with the International Rubber and Allied Trades Exhibition in London, Frank (18)



Rubber World

Fig. 1.—Hand operated Delaloe machine, as reproduced from "Science of Rubber" by Memmler, American edition, 1934. (Reinhold Publishing Corp., New York, N. Y.)



Rubber World

Fig. 2.—An early Schopper Dalen tension machine. Load was applied by hydraulic pressure from water main directly connected to the apparatus. (From an early catalogue by Louis Schopper, Leipzig, Germany.)

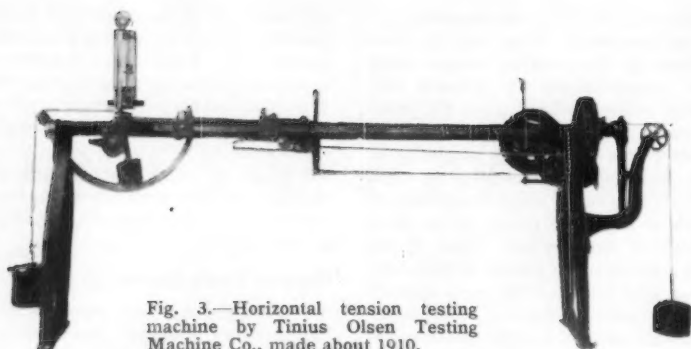


Fig. 3.—Horizontal tension testing machine by Tinius Olsen Testing Machine Co., made about 1910.

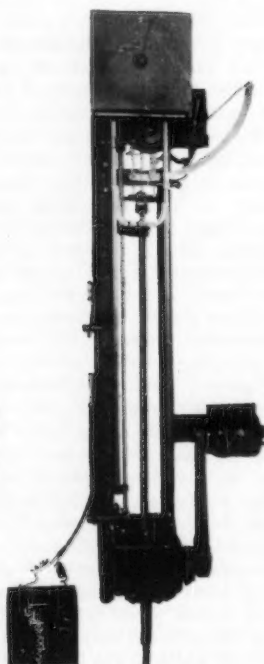
Rubber World



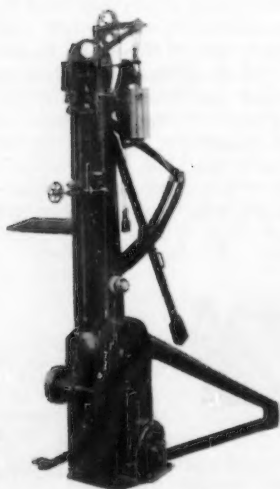
Rubber World

Fig. 4.—Motor-driven, spring-type machine developed at the Bureau of Standards in 1912.

Fig. 5.—One of the early tension machines of Henry L. Scott & Co. (now Scott Testers, Inc.)



Rubber World



Rubber World

Fig. 6.—The vertical Olsen machine, long known as the "Navy Tester."

described the new Schopper-Dalen testing machine (Fig. 2). Interest in testing at this meeting was such that it was decided to organize an International India-rubber Testing Committee. The next spring, as a member of the British Section of this committee, Schidrowitz (12) published an article on "The Tensile Properties of India Rubber" in which he discussed the earlier work and equipment and suggested the desirability of standardizing in certain respects some of the physical tests. He also said that the new Schopper machine was the "only one which to his knowledge had been designed solely for work with rubber."

About this time Memmler and Schob (19) started reporting their carefully planned large-scale studies of mechanical testing at the Staatliche Materialprüfungsamt in Berlin-Dahlem and discussed the effects of many variables. They used the Schopper machine for tension tests. The first American tension tester designed for rubber to be reported in the literature was a horizontal machine (Fig. 3) described by Olsen (20) at a meeting of the American Society for Testing Materials in 1910. This machine was built at the request of Dr. Geer but apparently was never very widely used because the following year a vertical motor-driven spring-type dynamometer (21) was designed at the National Bureau of Standards and later made available commercially (Fig. 4). It was followed in 1912 by the Scott (22) pendulum machine (Fig. 5) developed from a fabric tester which had been in use for more than a decade. A vertical Olsen machine (23), long known as the "Navy Tester," (Fig. 6) which somewhat resembled the design of Schopper, became available later.

The rapid development of these new machines after so long a period of lethargy was probably due in part to increasing interest in the physical and mechanical properties of rubber, but it appears that the spreading system of specification purchasing with its need for quality definition which could be quickly and reliably measured exerted compelling pressure. As Lord Kelvin said "When you can measure what you are speaking about, and express it in numbers, you know something about it; but when you cannot measure it, when you cannot express it in numbers, your knowledge is of a meager and unsatisfactory kind: it may be the beginning of knowledge, but you have scarcely, in your thoughts, advanced to the stage of science."

The Need for Test Methods

The railroads had been purchasing materials to specifications for many

years and were extending this to rubber products. The Government agencies, too, were learning about rubber and preparing specifications. The need for means of acquainting suppliers with the requirements of materials in measurable terms and of assuring delivery as specified was being felt all through industry. This, of course, necessitated development of equipment and methods of test which could be used and duplicated in different laboratories in the effort to be "of one language and one speech." Also, the increasing use of organic accelerators in rubber compounds was rendering chemical methods less indicative of the quality of vulcanized rubber with consequent turning toward physical tests.

In the summer of 1911, the International Testing Committee met in London in connection with the 2nd International Rubber and Allied Trades Exhibition. It is noteworthy that in opening the meeting, Dr. A. H. Berkhout (24), who had been Conservator of Forests of Java, said, "First of all, we shall have to attack the difficult problem of establishing standard methods. It is, of course, necessary we should agree in regard to the physical and chemical methods to be employed in testing india-rubber. It is not as it should be when a man can see the significance of his own figures but is not able to understand somebody else's." Little actual standardization was accomplished at that meeting, but an extensive outline was prepared of tests to be considered. These included stress-strain to break, effect of repeated stretching, tests of abrasion and hardness, effects of heat, light, water, oil, acid, and alkali, permeability to gases, electrical tests, and chemical analysis. That same year, at the Third International Rubber Congress in London, Memmler (25) gave detailed information on the mechanical tests of rubber and the equipment used at Berlin-Dahlem. He described tests of permanent set, aging, and folding resistance as well as the Martens' crushing test, Mai abrasion test, Brinell indentation, Schopper pendulum impact, and others.

Later, that year, an anonymous writer (26) in the *India Rubber World* commented, "It is worthy of remark that of late years, the technique of the rubber industry has ceased to rely solely upon data empirically or accidentally found. . . . In every quarter, inventive and creative brains have established mechanical tests, applicable to manufactured products or to those in process at the various plants. Consequently, these tests were from the very beginning diverse in character; varying from one laboratory to another,

both in the methods and the machinery employed. On the other hand, these tests are not sufficiently old to have attained uniformity through the adoption as standards of the best and most decisive amongst them. Nevertheless, the introduction of their standardization has been proposed by certain testing machine makers who, with a view to the solution of the problem, have produced testing machines which are intended to meet the requirements of the largest possible number of rubber manufacturers."

ASTM Committee D-11 Is Organized

The movement for standardization in this country culminated in December, 1911 in the organization of ASTM Committee D-11. It was significant that its first title was "On Standard Specifications for Rubber Products," which was later changed by deleting the reference to Standard Specifications. The committee consisted that first year of six rubber producers and fourteen nonproducers, of whom five represented railroads, two were Bureaus of the U. S. Navy, and three were from the electric industry. The objective was obviously to obtain assistance in the preparation of purchase specifications, for the first subcommittees appointed were on products such as hose, belting, packings, and insulating tape. The first ASTM rubber standards were completed in 1915 and were specifications and methods of test for fire hose and specifications for electrical wire and cable with insulation containing 30 per cent of Hevea rubber. The methods of test for hose, which included both chemical and physical tests, were intended to be revised to cover additional rubber products from time to time as specifications for them were completed. However, these methods remained without major change for seven years while the committee worked almost exclusively on product specifications, practically all of which were for goods used either by the railroads or the electric industry.

Meanwhile, in 1912, the National Bureau of Standards published the first edition of its *Circular 38* entitled "The Testing of Mechanical Rubber Goods" describing the equipment and methods employed at the Bureau. The tests covered were tension tests using the new Bureau of Standards dynamometer, elasticity or permanent set using a special extension table, reduction in tension when stretched to a definite elongation (stress decay) and "friction pull" (ply adhesion) by the dead weight method.

The apparatus was all either designed or adapted by P. L. Wormeley (27)

who in 1914 presented a paper at the Fourth International Rubber Congress at London on the "Influence of Temperature on the Tensile Properties of Rubber Compounds." In this paper, Wormeley mentioned the use of the above tests in specifications in the United States and the variations in each of them resulting from testing at different temperatures. He pointed out the need for standardization, the object of which, he pertinently remarked, "should be to make the work of different laboratories comparable." Thus he implied the confusion which was to grow worse before improvement came.

Variations in results were unavoidable when people were just learning the effects of the many variables in test conditions, but as laboratory workers became familiar with them the less scrupulous sometimes used them to their own advantage. Before temperature of testing was specified, a passing hardness value, for example, could often be obtained by immersing soft specimens in cold water for a time. I am reminded also of the early worker whose job required testing of tensile strength of specification goods using a small hand-operated screw machine. When his results were low, he turned the hand-wheel faster. In commenting on this, he said, "I got so I could play that machine like a violin." Nevertheless, the variations encountered in ordinary everyday testing, carried out with what was thought to be proper care, were the source of endless difficulty.

By the end of World War I, testing problems had become so annoying that various agencies began to give them serious attention. In 1920 a subcommittee on Standard Procedure for Testing Rubber Products was authorized by ASTM Committee D-11 and work was started on general methods of physical testing to include and replace the old fire hose methods. Shortly thereafter, the National Bureau of Standards advised the committee that a project was being started there on rubber. The committee suggested that first consideration be given to an accelerated aging test and appointed a special subcommittee to cooperate. Also, a new subcommittee on chemical analysis was organized to take over the work of the Joint Rubber Insulation Committee which had been concerned with chemical methods for a period of about seven years. At the same time a committee on methods of analysis was appointed by the Division of Rubber Chemistry of the American Chemical Society which had been officially organized in 1919. The chairmen of these committees, Simon Collier (28) for the Rubber Division and C. R.

Boggs for Committee D-11, held a joint meeting in Pittsburgh in 1922 and agreed on a draft of standard methods of chemical analysis of rubber products. These methods were eventually approved and published by ASTM and have since stood with comparatively few revisions as a landmark of standardization in rubber technology.

Performance Tests Appear

At about this same time, Committee D-11 formed a subcommittee on performance tests for which work on abrasion, aging, and oil-resistance was planned. Flex-life of belting was added later. Meanwhile, the Division of Rubber Chemistry had appointed a committee on physical tests with W. W. Vogt as chairman. Its report (29) to the Division in April, 1924 was a masterful presentation of the methods of preparing and testing experimental vulcanizates by the so-called tension tests, including discussion and experimental data on many of the variables requiring standardization.

In 1926, a new enlarged physical testing committee was appointed to investigate the effect of variables such as temperature and relative humidity upon the physical properties of rubber. This committee chose the problem of determining the importance of controlling atmospheric temperature and relative humidity while conditioning rubber test specimens at various stages of preparation and testing. The work was carried out at the National Bureau of Standards by F. B. Rupert as a research associate under the direction of the committee. A report (29) published in 1928 dealt with stress-strain and tensile properties and another (29) the following year with abrasion resistance. Finally, in 1930, the committee published a recommended outline of a tentative standard procedure for the preparation and physical testing of rubber (29). At that time the suggestion was made by J. W. Schade (30), but was not followed, that the committee extend the work by having a representative go to various laboratories in the industry and apply the principles of standardization in the recommended procedure to try to eliminate the conflicting results often obtained on the same material tested in different laboratories.

Many probably remember the tremendous variations that were common in the results of physical tests when the Rubber Division was sponsoring this work. These were particularly pronounced with compounds of the pure gum type. In a statistical study of stress-strain tests of 94 test specimens cut from a single factory inner tube,

Wiegand and Braendle (31) reported tensile strength results ranging from extremes of 2220 to 2950 psi with a distribution curve skewed toward the higher value.

When tests were made in different laboratories, the results were worse as was shown in a study of aging tests of three compounds in eight laboratories conducted by an ASTM subcommittee. The test sheets were compounded, mixed, and cured in one laboratory and, in all, over 5000 dumbbells were pulled. When, after three years, the work was completed, the subcommittee was unable to report any definite conclusions because of the variations obtained from both the original and aged specimens. By that time the ACS procedure had become available, and the subcommittee repeated the original test of one compound in thirteen laboratories. In reporting this series, the chairman, R. A. Schatzel (32) stated that whereas the previous variation in tensile strength had been between 2920 and 4144 psi, the new one was between 3232 and 3792 psi. The maximum variation of all laboratories, checked against the average tensile strength, was approximately 6 per cent. A similar value was obtained for the moduli at 400 and 600 per cent, while ultimate elongation gave much closer agreement. Similar improvement from standardization on the ACS die and controlling atmospheric conditions was reported by W. L. Sturtevant (33) in a series of tests in five laboratories.

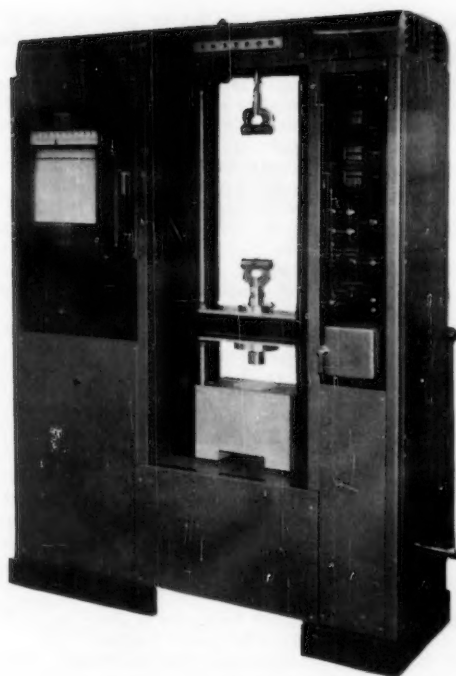
During the period of the great contributions by the Rubber Division, ASTM Committee D-11 was undergoing change. The need for better tests was increasingly realized, and dissatisfaction with some of the existing product specifications grew, both because of lack of agreement in the test results required for their enforcement and because it was felt that they failed to give adequate assurance of serviceability in many industrial applications. Consequently, the Committee decided to concentrate on development and standardization of test methods, leaving the question of detailed purchase specifications for reconsideration after establishment of suitable standard methods of test. With this change in objective, progress became rapid. New subcommittees were formed which studied and prepared many new test procedures, obsolete specifications were discontinued, and technical papers on rubber properties and testing began to appear in ASTM publications. The Rubber Division standard procedure for physical testing was incorporated so far as possible in ASTM methods. Finally, in 1935, the first of the special

compilations of ASTM Standards on Rubber Products (34) was issued. This book, presenting in consolidated form the results of many years of effort by numerous workers in the field of voluntary standardization of chemical, physical, and electrical testing of rubber products, soon became an indispensable laboratory manual as well as a reference source for specification writers. It has since been published on an annual basis, incorporating the results of new developments and changes agreed upon in the parent committee until it is now a substantial volume of more than 800 pages having very wide acceptance in the industry.

With the achievement of a considerable degree of standardization in rubber testing by agreement after the long years of research for new knowledge and of development of new methods and test equipment, there still remained the problem of having the individual laboratories apply the standard methods with the care and meticulous attention to detail that is necessary for success in test work. Troublesome variations still occurred which were often shown to be caused by some small detail or lack of refinement of equipment which had been overlooked. In other cases, existing knowledge concerning the test or the characteristic being measured was not sufficient to permit the accuracy desired. Important contributions in this phase of the testing problem have been made by the Joint ASTM-SAE Technical Committee on Automotive Rubber organized by the two societies in 1939 as a cooperative meeting place for automotive engineers and rubber technologists. In addition to outstanding work on rubber testing, this committee has developed specifications (35) for automotive rubber which have greatly simplified purchasing practices in the industry.

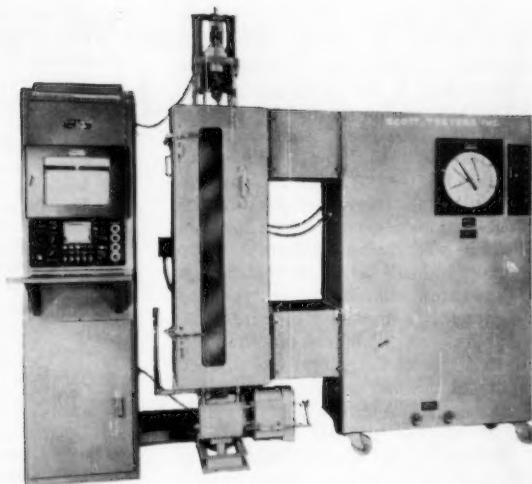
Standardization Matures in Synthetic Program

The culmination came in the synthetic rubber program of World War II when, of necessity, standardization reached the highest level yet attained in the rubber industry. The story of this program has often been told. It was realized from the beginning that the plants and their operation, the processes and the testing of the product would require the utmost in standardization to produce uniform rubber at so many locations and every effort was made to assure that result. I do not need to mention, for example, the adoption of GR-S for the greater part of the production, the design of the standard plant, or the precautions that were taken to assure uniform raw materials.



Rubber World

Fig. 7.—Instron Engineering Corp. floor model with load capacities from 2 g to 10,000 lb.



Rubber World

Fig. 8.—Scott Testers machine equipped with Accr-O-Meter electrical weighing and recording system, and high- and low-temperature condition box and test chamber.

These and many other phases of the program, including the standardization of manufacturing processes and of testing controls both during manufacture and on the finished product, have been discussed in detail by Livingston and Cox (36) in their very thorough chapter on "The Manufacture of GR-S" in the book "Synthetic Rubber" published under the auspices of our Division of Rubber Chemistry. I only wish to emphasize their statement that "Before 1943, the accuracy of the physical testing of vulcanized rubber by the rubber industry left much to be desired; systematic errors were found in most physical testing laboratories, and there was considerable variation between different laboratories." They further state that in consequence of an intensive drive to improve the precision of laboratory testing, the standard deviations of the test results obtained in the various copolymer plants in 1948 had been reduced to a level only one-half to one-sixth that of 1943.

This drive for reduced variability of test results was carried out on many fronts (37). A standard reference laboratory was built in Akron. Uniform lots of laboratory compounding ingredients were established and standardized by the National Bureau of Standards. Daily testing of reference lots of standard rubber by each laboratory was required. Testing equipment was standardized and improved by the National Bureau of Standards and the testing machine

manufacturers. Examples are the modification of the Mooney Viscometer (38) and the ORR L-5 Model of the Scott tester (39). Finally, an educational program like that suggested by Schade to the Physical Testing Committee in 1928 was adopted, and Dr. Garvey, present chairman of the Rubber Division, then with the Office of Rubber Reserve, and Dr. Stiehler, of the National Bureau of Standards, visited each laboratory and assisted in training all technicians to perform the tests by the same standard procedure. In standardization of testing it is indeed necessary to follow the admonition of Pasteur (40) to "Exhaust every combination, until the mind can conceive no others possible."

Of recent years, the rubber literature covering the investigations of physical and mechanical behavior of the material and the new methods and equipment for measuring them has grown to such proportions that even the most important contributions cannot be mentioned here. During the past two decades, a large proportion of these developments has resulted from the stimulus of the synthetic rubber program and has been surveyed by A. E. Juve (41) in his excellent chapter in the book "Synthetic Rubber" referred to previously.

The Future—More Work

Despite the great progress made, there is still much that remains to be done. Too little is known about the complex

properties such as abrasion resistance, adhesiveness, and tear resistance. Fundamental scientific work is needed on the precise mechanism of these phenomena and their relation to the intimate structure of the material, before good methods of controlling and measuring them can be developed. The tests presently used are undoubtedly measuring something accurately, but we are not sure just what, and when they fail to evaluate serviceability it is doubtless because the test and the service involve different conditions of which we do not appreciate the significance, if indeed we are aware of them at all. When we become annoyed at difficulties of this sort, let us consider how badly we would be handicapped in rubber manufacture if we had to do without our magnificent laboratories with their wealth of equipment and the years of research which have produced them and go back only half way to the time of Charles Goodyear when the only means of judging quality and serviceability was to try an article in the intended service.

Unfortunately, research in physical testing does not appear to offer the prospect of immediate financial return, and it is therefore often difficult to obtain for it the necessary support both in manpower and money. Yet history provides ample evidence that the development of new methods of observation and measurement is an indispensable forerunner of many of the greatest discoveries. The X-ray opened the

way to our present knowledge of the crystallization of rubber (42). The electronic vacuum tube gave us radio and television. The electrical resistance strain gage combined with the vacuum tube and cathode-ray oscilloscope has made possible not only better rubber testing machines (43, 44) (Figs. 7, 8) but also the studies of stresses and vibration under conditions of service which have resulted in safer, more comfortable automobiles and lighter, better airplanes. The electron microscope has revealed new worlds in the field of reinforcing pigments. As a final recent example, infrared spectrometry (45) has enabled us to know when we had produced a synthetic rubber that is really a duplication of the natural product. It is interesting to wonder how many laboratory workers on synthetic rubber in the past may have made this material in their mixtures without realizing its existence because they had no way of identifying it. Truly, in the words of Pasteur (46), "All things are hidden, obscure and debatable if the cause of the phenomena be unknown, but everything is clear if this cause be known." We must devote more attention to causes.

We are now on the threshold of a new age—that of the utilization of atomic energy. Tires have already been vulcanized by the rays of this source of energy. Who knows what great events lie ahead? Two years ago Dr. Dinsmore (47) told us about the depth and breadth of knowledge required today of a rubber technologist. Again, we are reminded of the words of Pasteur who said "The Sciences gain by mutual support," and, "It is characteristic of science to reduce incessantly the number of unexplained phenomena" (48). While we cannot now see the future, it is clear that we have in the industry the depth and breadth of knowledge in all of the sciences that will be required to meet the challenge to the end that the unexplained shall disappear. In the words of Michael Faraday (49), "In the pursuit of science, we first start with hopes and expectations; these we realize and establish, never again to be lost, and upon them we found new expectations of farther discoveries, and so go on pursuing, realizing, establishing and founding new hopes, again and again." Perhaps, at last, we shall rebuild the Tower of Babel which will reach unto the Gate of the God.

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Analysis of Radioactivity in Surface Waters— Practical Laboratory Methods

By L. R. SETTER, G. R. HAGEE, AND C. P. STRAUB

RADIOACTIVITY was first observed in 1896 by Henri Becquerel who demonstrated that uranium would fog photographic film. Shortly thereafter, it was learned that thorium and actinium were also radioactive and that there were long series of radioactive substances associated with each of these three elements. It was found that the parent elements changed successively from element to element by the emission of alpha, beta, and gamma radiations until some stable end-element was produced; in the case of these three series, the stable end-element was an isotope of lead. These naturally occurring series elements, through their radioactive daughter gases radon and thoron, have an appreciable effect upon background or air activity. These activities are washed down with rain and are carried into water supplies.

In addition to the radioactive elements forming the series mentioned above there are other naturally radioactive elements. Those listed by Cowan (1)¹ include potassium-40, rubidium-87, samarium-147, lutetium-176, and rhenium-187. Of these, the radioactive potassium is of most interest. It is found everywhere and amounts to 0.011 per cent of the natural element. The amount of potassium 40 in the oceans is estimated to be 5×10^{11} curies or about 300 μc per liter (1).

With the development and operation of nuclear reactors and other types of atom smashing machines, large quantities of radioactive elements are produced. These include all the elements in the periodic table.

Since radioactive materials are damaging to tissue, standards have been established for their control. The Atomic Energy Commission specifies that the level of external and internal

exposure for its workers should not exceed 300 mr per week. For the general population it has been suggested (2) that this value be reduced by a factor of 10. The National Committee on Radiation Protection (3) and the International Commission on Radiological Protection (2) have proposed maximum permissible concentrations for many of the radionuclides in drinking water and in air. It is recognized that these values for evaluating the radioactive hazard are predicated upon incomplete biological data and may change as newer information becomes available. For unknown mixtures of radioactive materials the recommended permissible concentration in drinking water for alpha, beta, or gamma contaminants is 100 μc per liter,² this value also being subject to reduction by a factor 10 when applied to the general population.

Should the activity of the drinking water exceed 100 μc per liter it would be necessary to analyze the activity for specific radionuclides to evaluate the hazard. The two most hazardous radionuclides in so far as drinking water is concerned are radium (an alpha emitter) and its daughter products, and strontium-90 (a beta emitter). Proof that negligible quantities of these radionuclides were present in drinking water would permit raising the permissible level to 1000 μc per liter or more, even though the basic philosophy is to keep the levels of radioactivity as low as possible.

When released into the environment, certain radionuclides concentrate in bottom muds, soils, and biological materials. Accordingly, analyses are required to determine the degree of such concentration and, if necessary, the

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NOTE.—DISCUSSION OF THIS PAPER IS INVITED, either for publication or for the attention of the author. Address all communications to ASTM Headquarters, 1916 Race St., Philadelphia 3, Pa.

¹ The boldface numbers in parentheses refer to the list of references appended to this paper.

² 1 micromicrocurie (μc) = 1×10^{-12} curies.

amounts of activity contributed by specific radionuclides. The purpose of this paper is to present analytical methods for the determination of gross alpha and gross beta radioactivity in water, soil, and biological materials, as developed at the Robert A. Taft Sanitary Engineering Center.

Samples and Sampling Procedures

In sanitary science the alpha and beta-gamma radioactivities of snow, rain, and surface or ground waters are of importance as they affect the quality of water supplies and of plant and animal foods. The water-soluble fraction is of more immediate concern in water supplies where treatment by chemical coagulation and filtration is used to remove insoluble or suspended matter.

The radioactivity of soils, river bottom sediments, and biological materials is also of interest, because many isotopes tend to become fixed on air particulates, soil particles, vegetation, plankton, etc., and may reveal an accumulation or concentration of activity not detectable in waters.

Standard procedures (4) are usually satisfactory for the selection of sample containers and the collection of representative samples. When highly acidic or alkaline wastes are being assayed, control tests may reveal the deposition of radioactivity on container surfaces. The time interval between collection and analysis is important if levels of short-lived activity are sought or if they are present in amounts which will interfere with the analysis of longer-lived isotopes. For example, in samples of air particulates the radon daughters (composite half-life of 36 min) and thoron daughters (composite half-life of 10.6 hr) can each be estimated if the samples are alpha counted within the first half-hour of collection and again after 6 to 12 hr. The longer-lived mixed fission products may be determined by beta counting samples two or more days after sample collection (to permit decay of radon and thoron daughters) or by counting both the alpha and beta activity for an earlier assay. The beta activity of radon and thoron daughters may be calculated from the alpha results

and deducted from the gross beta activity for residual beta activity. Prolonged storage of samples for weeks will reduce through decay the sensitivity of the tests and introduce errors of extrapolation from decay data to the time of collection. Since the distribution of dissolved and suspended activity may change with storage, separation by filtration should be made soon after sample collection. Sample preservation with acids or disinfectants is usually avoided because these agents may alter the distribution of activity as compared to unpreserved samples. Where used, it is always well to check the activity in the preserving medium along with a determination of activity in the solid fraction. Biological samples should be processed and composited in a fresh state where possible, then frozen or preserved if necessary for transportation and assay at some distant laboratory.

Sample Preparation

Although liquid and crystal scintillation counters warrant further study for assaying alpha, gamma, and perhaps some beta activity in solutions or slurries from original samples or concentrates, the conventional methods utilize internal-chamber or thin-window counters for measuring radioactivity. To be detected, the radiation must escape from the sample, enter into the sensitive volume of the counting chamber and ionize the counting gas.

The radiation from the most energetic alpha emitter and from beta radiation having an energy of 60 Kev will not escape from the sample if it is covered by a sample thickness of 8 mg per sq cm. The radiation from a weak alpha emitter will be stopped if covered by only 4 mg per sq cm of sample solids. Consequently, for low-level counting it is imperative to evaporate all moisture and preferable to destroy organic matter before depositing a thin film of sample solids from which radiation may readily enter the counter. Counting a thick sample is unreliable except for known mixtures of radioactivity.

With end-window counters having a diameter of less than 1 in. the sample is mounted on a smaller diameter planchet or dish and counted some distance below the window. Radiation losses occur in the air space, the window, and the insensitive volume of the Geiger-Müller tube. The farther the sample is placed from the window the lower the geometry or percentage of radiation (emitted in all directions) entering the counter. It is because of the improved geometry and relatively low self-absorption losses which occur that the Robert A. Taft Sanitary Engineering Center

has adopted internal chambers for the alpha and beta counting of samples in dishes 2 in. in diameter.

Water Samples

Since it is desirable to differentiate between radioactivity associated with suspended solids and that in solution, a volume of sample (usually 250 ml) is filtered through a membrane filter.³ The filter containing the suspended solids is dried (103 C), weighed, treated for counting, and counted preferably after thoron daughters have decayed to insignificant values.

Membrane filters are nonconductors which distort the electric field of internal counters and hence tend to suppress the true counting rate. This count suppression is energy dependent. It is however a variable quantity for samples of the same radionuclide, and hence a direct correction for it cannot be made. For example, it has been found for zirconium-95 ($E_{\max} = 0.37$ Mev)–columbium-95 ($E_{\max} = 0.16$ Mev) the suppression varies from 10 to 40 per cent of the true counting rate. The charge problem is overcome readily by (a) wetting the dry filter with a minimal quantity of conducting fluid⁴ followed by drying and counting, or (b) saturating the filter with ethyl alcohol, igniting the alcohol, and finally directing the flame of a Meeker burner over the surface of the sample for 10 to 20 sec. Ignition without alcohol will result in violent burning with a resultant loss of sample. Either method (a) or (b) has been found to be dependable.⁵

The filtrate is transferred to a beaker and evaporated to dryness on a hot plate. The dry solids are acidified (one or two drops 1 N HNO_3 in excess of alkalinity) and transferred with a rubber policeman and distilled water to a weighed dish (aluminum), 48 mm in diameter, dried in an oven at 103 C, weighed, and counted.

Soil Samples

Sediments or soils low in organic matter are air or oven-dried and prepared for counting by the usual method of quartering for representative sampling. Several grams of fine mesh sample, ground in a mortar with pestle if necessary, are wetted with distilled water to make a thin cream. Quantities of sample roughly equivalent to 50, 100, and 200 mg of dry solids are spread to a uniform depth in tared dishes. The samples are then air- or oven-dried and weighed. Slow drying of clay samples usually results in a firm, continuous, and dustless deposition which permits counting without further treatment. Samples that shrink or crack or show evidence of loose dust are wetted with a

³ The tared weight of an aluminum dish (bottle cap) plus the air dry HA membrane filter 48 mm in diameter permits a rough estimate of the suspended solids and the transmission factor, since the suspended matter is deposited on an area 35 mm in diameter.

⁴ Many fluids are useful, such as Anstac C-A.

⁵ For samples of air particulates collected on glass fiber filters Anstac M has been found satisfactory, but the burning technique applied to membrane samples is simple, less time-consuming, and permits immediate counting for radon daughter activity.

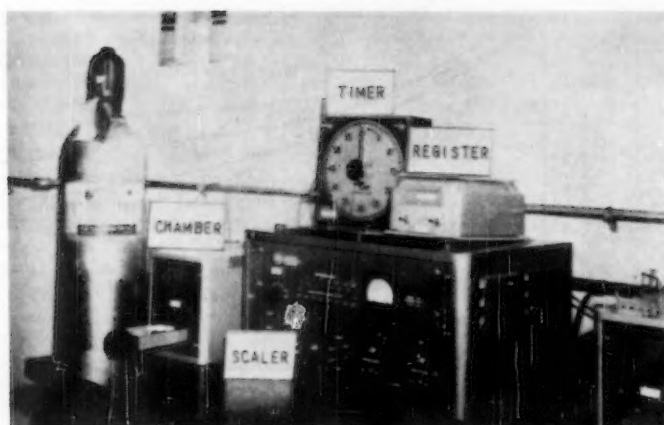


Fig. 1.—Internal proportional counter.

few drops of acetone containing 0.5 per cent lucite. The acetone evaporates leaving enough Lucite binder (2 to 5 mg) to hold the sample firmly and dust-free while counting. Sediments high in organic matter are dried at 103 C and ignited at 600 C prior to distributing suitable aliquots of fixed solids on dishes as above.

Biological Samples

Organic matter such as leaves, grasses, algae, fish bones, or scales, and similar fat-free matter is prepared for counting by determining the dry solids (103 C) and fixed solids (600 C) of large representative samples (1 to 20 g of fixed solids) in silica or stainless steel dishes. Being corrosive to aluminum, the fixed solids are usually neutralized with 1 N HNO_3 and then kneaded with a glass rod or pestle to form a smooth thin cream similar to that made in preparing sediment samples. Suitable aliquots of the moist sample are transferred to counting dishes. The samples are oven dried, weighed, treated with acetone-Lucite if necessary, and counted. Small samples containing less than 300 mg of fixed solids are processed directly in several stainless steel dishes having a diameter of 2 in. and a capacity of 10 ml.

Some biological samples such as milk and muscle tissues cause difficulty on ignition and are best processed for counting by wet ashing. Two- to 10-g samples of moist tissue are placed in a small (50 to 100 ml) beaker and digested for periods of from 10 min to overnight at room temperature with approximately 5 ml of concentrated HNO_3 (specific gravity 1.42). Following solution, the samples are warmed gently (hot plate) with increasing heat and evaporated to dryness. After cooling, 5 ml concentrated HNO_3 is added to each

digestate for a second and a third acid evaporation to dryness. One ml of concentrated HNO_3 and a few drops of hydrogen peroxide (30 per cent) are used for the fourth digestion. Except for a globule or two of fat in some samples, the digestion should be complete. Residual acid is removed with two successive evaporations of distilled water used to wash down the sides of the beaker. For small samples containing less than 50 mg of ash the entire contents of the beaker are transferred with distilled water and a rubber policeman to a counting dish. The sample is evaporated to dryness in an oven (103 C), weighed, and counted. For large samples such as bone, the digestion is carried out in a weighed beaker, and after digestion the ash is diluted to a known weight with

distilled water and appropriate aliquot volumes are transferred to counting dishes. The samples are evaporated, oven-dried, weighed, and counted. If they appear dusty they are treated with acetone-Lucite to prevent counting difficulties and loss of sample.

Radioactivity Measurements

The radiation counter (Fig. 1) consists of a methane-argon flowtype counting chamber (Fig. 2), a pre-amplifier, and an electronic scaler and power supply assembly. An individual radioactive emission initiates ionization of gas which is detected by the counter. The scaler totals the ionization events occurring in a selected time interval.

By proper adjustment of operating voltage, both alpha and beta activities may be measured in internal proportional counters. At the low voltage suitable for alpha counting, beta emissions are not detected because the counting gas is insufficiently ionized to activate the pre-amplifier. When the voltage is adjusted to a higher value suitable for counting beta activity, both beta and alpha radiations activate the pre-amplifier.

Voltage Plateaus

The criterion for counter stability and reproducibility is that a plot of counting rate versus instrument operating voltage should possess a broad and flat plateau region. This characteristic is influenced by the area of the source and the size and position of the high-voltage electrode (5). The plateaus are determined with standard radioactive sources which have areas comparable to those of the count-

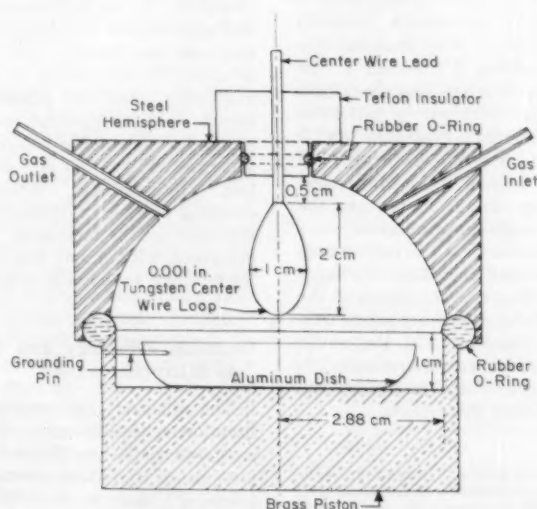


Fig. 2.—Internal counter chamber.

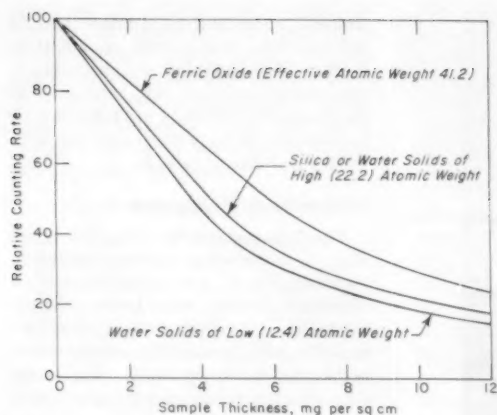


Fig. 3.—Self-absorption of natural uranium alpha activity.

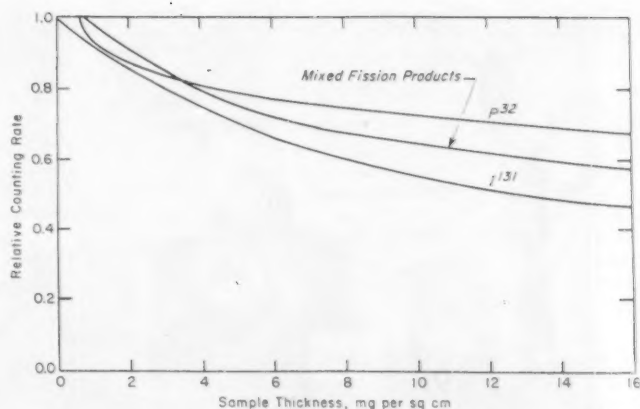


Fig. 4.—Self-absorption in an internal counter.

ing dishes. The instrument will perform satisfactorily at the mid-plateau voltage if the plateau is 150 v or more in length with a slope of less than 3 per cent per 100 v.

Calibration Standards

A standard source (secondary standard) of uranium oxide plated on stainless steel is used to determine alpha voltage plateaus and check instrument performance. A similar standard source covered with 8 mg per sq cm of aluminum foil (which completely attenuates alpha activity) is used to determine beta plateaus and serve as a permanent beta standard. Periodically the instruments and the permanent standards are checked against certified standards.⁶

Stability of counter performance is checked with a permanent standard three times a day. Any counting-rate deviation exceeding 2 per cent of the standard value at a counting confidence limit of 95 per cent (6) is indicative of improper instrument behavior, and if minor adjustment in the operating voltage does not correct the performance, other steps are taken to remedy the difficulty. A high background indicates chamber contamination. Erratic counting may be due to high voltage discharge from dust or excessive roughness of the electrode. The preamplifier tubes, condensers and resistors, and the scaler tubes and circuitry are then checked in the listed order to locate the trouble and make appropriate corrections.

⁶ Obtained from National Bureau of Standards. Two types of standards are employed: (1) Radium DEF on silver; (2) solutions of specific radionuclides such as sodium-24, phosphorus-32, and iodine-131. Aliquots of the solutions are evaporated in counting dishes and measured.

Instrument Background

The background of alpha radiation is usually less than 0.5 count per min unless the chamber has become contaminated. Cosmic and gamma radiation, however, cause a background of 50 to 60 counts per min at the voltage used in counting beta activity. Background measurements are made on empty counting dishes three times daily for two 8- and one 16-min intervals following instrument calibration with the standard. This 32-min total counting period per day yields a value for alpha background usually reliable to ± 50 per cent and for beta activity ± 5 per cent.

Samples are arbitrarily counted for two 16-min periods, each preceded by a 30- to 60-sec gas flush of the counting chamber. If the two measurements do not agree within counting statistics (6), improper gas flushing is suspected and a third 16-min count is made immediately. For this practical time of sample and background counting, the minimum detectable alpha and beta activities (7) are 0.5 and 4 counts per min, respectively. Where greater counting precision is desired, the counting time can be increased. It may be decreased with minor loss of precision for samples of moderate to high activity.

Counting Efficiency and Conversion to Micromicrocuries

The efficiency of counting, E , is a function of the geometry, G , the backscattered radiation, B , and the transmission of radiation through sample solids, T ; that is, $E = GBT$. For the type of internal counters used, the geometry factor is 50 per cent or $G = 0.5$.

Backscatter

In measuring alpha activity, very little radiation is backscattered or reflected into the counter from the sample dish. At most this amounts to 2 per cent, or the backscatter factor $B = 1.00$ to 1.02. The backscatter factor for beta activity is dependent on the energy of the radiation and the effective atomic number of the sample dish and its support. Two-and one-half-year old mixed fission products and thallium-204 standards in aluminum dishes mounted in a brass chamber have a backscatter factor of 1.29 and 1.31, respectively (13).

Self-Absorption

The transmission of radiation through sample solids is a function of amount and chemical composition of solids, the type of radiation, and its energy. For alpha activity of natural uranium (energy of 4.2 and 4.76 Mev) the transmission is seriously reduced (8) by a sample thickness of a few mg per sq cm as shown in Fig. 3. This figure also shows that an increase in the effective atomic weight of water solids, such as would result from a high chloride content, permits greater transmission for samples of equal thickness. The alpha transmission in soils having 100 per cent silica (effective atomic weight of 21) is substantially less than in ferric oxide (effective atomic weight 41.8). When testing alpha activity of unknown energy in solids of unknown composition, it appears that samples of minimum thickness (less than 4 mg per sq cm) should be counted to avoid uncertain corrections for transmission.

The loss of transmitting beta activity through sample solids is less than for alpha activity, but nevertheless sample thickness has an important influence on the true counting rate as shown in Fig. 4.

The figure shows the transmission through sample solids for phosphorus-32 (maximum energy, 1.7 Mev), for iodine-131 (maximum energy of largest component, 0.6 Mev), and for one-year-old mixed fission products.

The transmission of activity from fission products will vary somewhat with the age of the sample. In correcting for beta transmission, it is assumed that unknown beta emitters have energies comparable to one-year-old mixed fission products.

The conversion to curie units of the observed counting rates of samples measured in counters of known efficiencies involves dividing the counts per min by 2.22E where 2.22 disintegrations per minute is $1 \mu\text{C}$ (10^{-12} curies) and E is the fractional efficiency of the counting arrangement.

Interpretation

The above methods of radioactivity analysis have been applied to surface and ground waters throughout the United States over a period of six years with but minor changes in technique. The levels vary depending on the natural radioactivity and man-made pollution. Excluding the environmental area near reactor sites, air-borne fission products from the testing of nuclear weapons have been largely responsible for the increase in radioactivity shown in Table I.

The table lists the ranges of activity found in the Cincinnati, Ohio, air and rain in 1954 and 1956 (primarily due to the testing of nuclear devices in the Pacific) as compared to that in cistern water, on leaves of trees near a Cincinnati pond, in the pond water, and in plankton, fish, and mud of the pond. Usually the air and rain activity could be identified from decay data as being fission products formed on a specific test date and having a negative exponential decay factor of 1.2. The rapid decay of young fission products, physical accretion of activity on surfaces, sedimentation, and dilution all account for a reduction of radioactivity in cistern, surface, and pond water as compared to that found in rain.

Rain samples if processed and measured for radioactivity within 1 to 2 hr after collection have been found to contain significant quantities of radon and thoron daughter products. In such cases it is necessary either to (1) make both alpha and beta measurements and correct for the radon-thoron daughter content or (2) measure activities after these constituents have decayed to insignificance. It is pertinent to observe (Table I) that the range of radon-222 activity in Cincinnati air is many times higher than that of mixed fission products.

TABLE I.—ACTIVITY LEVELS IN VARIOUS MEDIA, CINCINNATI, OHIO.

Medium	Year	Unit	Gross Alpha		Gross Beta	
			Suspended	Filtrable	Suspended	Filtrable
Rainfall.....	1954	μC per liter	29- 2 250	52- 5 050
	1956	μC per liter	27-24 000	44-16 500
Cistern water.....	1956	μC per liter	13- 1 330	34- 1 500
Ohio River.....	1956	μC per liter	<1.5	<3-3	<10- 82	<10- 60
Tap water.....	1956	μC per liter	<10- 22	<10- 22
Pond.....	1954	μC per liter	20-280
Air.....	1954	μC per cu m	0.01-17
	1956	μC per cu m	280-3500 ^a	0.4-9
	1956	μC per cu m	8-33 ^b
Leaves.....	1954	μC per g ^c	120-1500
Plankton in Ohio pond..	1954	μC per g ^c	40-5100
Fish in Ohio pond.....	1954	μC per g ^c	0-31	110-260
Bottom mud of Ohio pond.....	1954	μC per g ^c	50 -200

^a Radon-222 in Oct., 1956 samples.

^b Alpha activity from daughters of lead-212 (thorium B) in Oct. 1956 samples.

^c Of fixed solids.

Discussion

Internal proportional counters have been selected for low-level radioactivity measurements because they possess a high, constant, and reproducible geometry or efficiency. End-window Geiger-Müller and crystal scintillation counters differ from these in that they have a low and variable over-all efficiency particularly for measurements of weak energy beta radiations. Hence, great uniformity of sample thickness is essential for reliable counting efficiency. Moreover, a second instrument is needed for alpha measurements.

In cases where it is possible to measure a radionuclide by means of its gamma radiation, crystal scintillation counters may be found useful because the penetrating power of gamma radiations make the measurement essentially independent of sample solids or media. Unfortunately, many radionuclides of interest (for example strontium-90) emit no gamma radiation or emit it in low abundance.

Liquid scintillation counters (9) appear to offer some advantages for counting both liquid and solid samples, but the techniques require further exploration. They may be found extremely useful both for gross measurements and for analyzing specific radionuclides.

Maximum permissible concentrations of radionuclides in water vary widely, and hence the hazard of contaminated water can be evaluated accurately only by specific isotope analyses. Certainly for sustained levels of activity near the maximum permissible levels further analysis is required—rapid decay in a sample may establish the absence of significant amounts of the more hazardous long-lived emitters. This is particularly true in water contaminated with young mixed fission products where a very large part of the total activity is associated with short-lived isotopes of low ingestion hazard.

Methods are available at present for determination of radium (10) and strontium (11, 12). Work is continuing on the development of chemical and physical methods for the quantitative analyses of other radionuclides.

Summary

Practical procedures for determining the gross alpha and gross beta radioactivity of water, soil, and biological samples have been described. The procedures consist of separating suspended activity, evaporation of filtrates, drying at 103 C or ignition at 600 C, and deposition of a thin film of sample solids on large (2-in. diam) dishes for counting in internal, gas flow, proportional counters having a maximum efficiency of about 50 per cent for alpha and 65 per cent for beta activity from one-year-old mixed fission products. The methods used are suitable for estimating nonvolatile radioactivity at levels far below $100 \mu\text{C}$ per liter (the maximum permissible concentration of unknown radionuclides in drinking water), are applicable at levels exceeding $100 \mu\text{C}$ per liter, and may through decay measurements or a knowledge of radionuclide composition, indicate the health significance of observed activity.

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Brittleness Temperature Testing of Elastomers and Plastics

By A. C. WEBBER

Subcommittee 27 on Low Temperature Testing of Elastomers and Plastics of ASTM Committee E-1 on Methods of Testing established, in 1951, a Task Group on Correlation of Test Methods. This task group, following a survey to determine tests of major interest for use at low temperatures, has studied methods of test for four properties: stiffness, hardness, rheological properties, and brittleness. The general pattern of study has been, first, to survey and select test methods for each property which are applicable at low temperatures and, second, to obtain test data by means of interlaboratory studies on the same group of elastomeric compounds. From these data, the task group has obtained information on the utility of various methods at low temperatures and plans to determine and report on any useful correlation which may exist among them. Reports of the work on the first three properties—stiffness, hardness, and rheological properties have been published (1, 2, 3).¹

THE TASK GROUP ON Correlation of Test Methods found only two methods in general use for the determination of brittleness at low temperatures, both ASTM tests.² Other possibilities were considered and, of these, two—a falling weight test and the Charpy impact test—were investigated briefly. Results of a round-robin study of the four test methods soon indicated that only one method among them, ASTM Method D 746-55 T, was useful over the range of materials tested. A recheck using

a modified criterion of failure was made. Results and conclusions of the task group are summarized in this report.

Definitions and Concepts of Brittleness

Early in the deliberations in the task group, this definition of brittleness was proposed: "the inability of a material to adjust itself to a dynamic stress." Further discussion by the task group indicated that this definition was deficient. For example, a plastic bar of a material considered brittle may be flexed repeatedly for millions of cycles at dynamic maximum stresses below its endurance limit, without fracture, because although brittle in the usual sense, it "adjusted itself" to these dynamic stresses. A rigid plastic, stressed to a fraction of its ultimate strength by a dead weight (a static load) may fail in time with a fracture characteristic of brittle impact failure, that is, a non-ductile, low-elongation break. Some flexible rubber compounds when struck

at room temperature by a rifle bullet exhibit cracking around the bullet hole, a typical indication of brittle failure. An elastomer is brittle whenever the rate of straining exceeds its relaxation rate.

In each of these examples, we are aware of brittleness only after a material has broken. It has been shown that neither hardness nor modulus of elasticity correlates fully with brittleness (5). Examination of the failure of brittle materials, or materials in a brittle condition, shows that the break always occurs at relatively low strain, with a characteristically smooth or conchoidal fracture surface. Usually crack propagation is evident in brittle failure. Because of the small strain, materials failing in a brittle fashion absorb less



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¹ The boldface numbers in parentheses refer to the list of references appended to this paper.

² Tentative Method of Test for Low-Temperature Brittleness of Rubber and Rubber-Like Materials (D 736-54 T), 1955 Book of ASTM Standards, Part 6, p. 1188; Tentative Method of Test for Brittleness Temperature of Plastics and Elastomers by Impact (D 746-55 T), 1955 Book of ASTM Standards, Part 6, p. 148.

energy per unit volume than tough materials; that is, less work is required to break the specimen. If we put these concepts together, a definition for brittleness might read "a material is brittle if, when stressed to fracture, it breaks with little strain, low energy absorption, and with a relatively smooth or conchoidal fracture surface or evidence of crack propagation."

It is well known that brittleness is not a fundamental property of a material but is dependent upon other variables in addition to the nature of the material itself. Two of the principal variables are temperature and rate of development of strain. It is common experience that at any selected rate of straining, there is a temperature or temperature range below which a material will exhibit brittleness and above which ductile failure will occur with high unit energy absorption relative to that of the brittle failure. Plots of specific volume, electrical loss, and various moduli *versus* temperature have shown that there are temperatures at which more or less abrupt changes occur in the slopes of the curves. Such a secondary transition region is characteristically associated with "brittleness temperature." For some materials, such as rubber, this temperature will be low and quite sharply defined. With others, like polyethylene, brittleness occurs over a wide range of low temperatures. For materials such as polystyrene, the brittle point may be well above normal room temperatures.

Ideally, the most useful information on brittleness could be obtained by determining a family of tensile stress-strain diagrams at rates from static to ballistic loading over a practical range of temperatures. Such information in the form of unit energy absorption *versus* strain rate and temperature would even be meaningful for design purposes. Unfortunately, no single piece of suitable test equipment is available for making these measurements, nor would the test be simple enough for general use if it were. For these reasons, simple tests have been devised to obtain approximate data, at convenient but high straining rates.

If we arbitrarily select a straining rate, or define a specimen and the loading parameters, then for these conditions a "brittleness temperature" may be determined. Provided that each material tested is equally sensitive to notches or

other stress-raisers, variations in straining rate, and the other minor variables in our procedure, we may compare and rank them according to their usefulness for applications comparable to our test conditions. The methods for determination of brittleness at low temperature which have been adopted by ASTM, and others which have been considered for this purpose, are based upon these concepts.

Test Methods

In the simpler methods for determination of brittleness temperature, at least three types of measurement are used:

1. Determination of a minimum temperature at which no failure occurs under specified straining rates;
2. Determination of the temperature at which 50 per cent of the specimens tested fail; and
3. Determination of the temperature at which a marked transition in some measurable property (such as electrical loss, specific volume, modulus of elasticity, or energy to break) occurs which might be demonstrated to correlate with the onset of the phenomenon of brittleness.

Methods using the techniques indicated in (3) above, while probably capable of providing quite exact data, were considered by the task group to be too complex and time consuming for routine use. Furthermore, no methods using these techniques have been standardized. Therefore, the following methods were studied:

1. ASTM Method D 736 (now revised) "Methods of Test for Low-Temperature Brittleness of Rubber and Rubber-like Materials".² In this test, specimens are die-cut in the form of die C of the familiar tension test ASTM Method D

412.³ Two specimens are placed bent in a loop between two horizontal plates separated $2\frac{1}{2}$ in. The fixtures and specimens are exposed to the test temperature for 5 hr. At the end of this period, the plates of the flexing fixture are moved rapidly, usually by hand impact at an unspecified rate, from the $2\frac{1}{2}$ in. separation to a position 1 in. apart. Cracks or fractures in the specimen are considered failure. If two specimens fail at the test temperature, the compound is considered to have failed.

2. ASTM Method D 746 (now revised) "Brittleness Temperature of Plastics and Elastomers by Impact."² In this method small specimens are clamped as cantilever beams in a horizontal position. After immersion in a liquid heat transfer medium at the test temperature for $3 \pm \frac{1}{2}$ min ($2\frac{1}{2} \pm \frac{1}{2}$ min in the 1952 version of Method D 746) the specimen or group of specimens is impacted by a standard striking edge at a speed of 6 to 7 ft per sec. Test specimens may either be die-cut pieces, 0.075 in. thick, $\frac{1}{4}$ in. wide or the familiar T-50 test specimen of ASTM Method D 599.⁴ The impact hammer may be energized either by a solenoid or by a motor in the commercially available form of the apparatus. In this method, ten specimens are tested at each of a sufficient number of temperatures to determine statistically the temperature at which 50 per cent fail. Failure was formerly specified in this method as complete separation of the specimen into two or more pieces. The present method alternatively defines failure as the appearance of any crack in the specimen which is visible to the unaided eye. Specimens A, B, C, and D in Fig. 1 illustrate the common types of failure. The 50 per cent

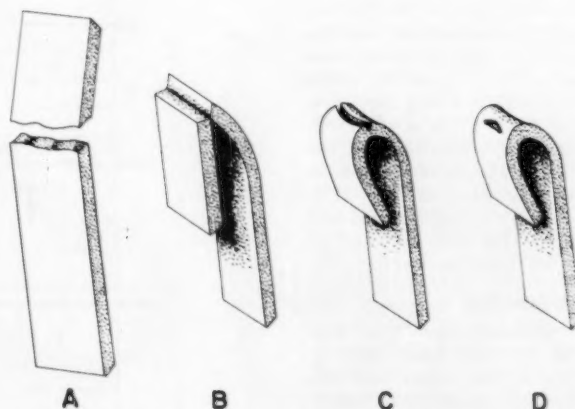


Fig. 1.—Types of failure in Method D 746 specimen.

A—complete separation C—partial break
B—"hinge" break D—crack or hole

² Tentative Method of Test for Tension Testing of Vulcanized Rubber (D 412-51 T), 1955 Book of ASTM Standards, Part 6, p. 1129.

⁴ Method of Test for Physical State of Cure of Vulcanized Rubber (T-50 Test), (D 599-55), 1955 Book of ASTM Standards, Part 6, p. 1072.

TABLE I.—MATERIALS USED.^a

A....	Gum rubber compound
B....	SBR (GR-S) compound, tread type
C....	Polyethylene
D....	Vinyl plastic, plasticized
E....	Neoprene

^a Materials A and B were prepared by B. G. Labbe, Government Laboratories, University of Akron; C and D by B. H. Carey, Bakelite Co., Bound Brook, N. J., and E by J. B. DeCoste, Bell Telephone Laboratories, Murray Hill, N. J.

TABLE II.

	Participant	ASTM Method	Apparatus
A..	American Cyanamid Co., Stamford, Conn.	D 746	Solenoid
C..	Bakelite Co., Bound Brook, N. J.	D 746	Motor
D..	E. I. du Pont de Nemours & Co., Inc., Polychemicals Dept., Wilmington, Del.	D 746	Motor
E..	Firestone Tire and Rubber Co., Akron, Ohio	D 736	...
G..	B. F. Goodrich Research Center, Akron, Ohio	D 746	Solenoid
I..	Rock Island Arsenal Ordnance, U.S. Dept. of the Army, Rock Island, Ill.	D 746 D 736	Solenoid Motor
K..	U.S. Rubber Co., Passaic, N. J.	D 746 D 736	Motor

brittleness temperature, T_b , may be determined by a formula (10,11) or by a graphical method. In addition to the standard determination of T_b , provisions have been made in the procedure for a routine acceptance test based upon a specification of a limiting percentage of failures at a specified temperature, or that none shall fail among five tested at a specified temperature.

3. Two other methods were considered and tried.

(a) *The falling weight (Tinius Olsen test)*

In this method, the test specimen supported as a simple beam, is loaded in impact through a knife edge at the center point by a 5-lb weight falling through a maximum of 3 ft. Tests at the U. S. Rubber Laboratory in Passaic, N. J., using this method in a conditioned low-temperature room, were inconclusive. Failures occurred only in one material and in no case were the data sufficient to determine a brittleness temperature.

(b) *Charpy Impact Test*

It was proposed also that this test, ASTM D 256, made over a series of temperatures, might show the point at which the transition to brittle failure occurred. This method was explored at the Armstrong Cork Co. Since only the vinyl specimens could be broken at the lowest available test

TABLE III.—BRITTLINESS TEMPERATURE DATA (T_b deg C).

Method D 746	Laboratory	A Gum Rubber	B SBR (GR-S)	C Polyethylene	D Vinyl
Solenoid.....	A	-52	-71.4	-62.5	-31.2
	G	-53	-63.6	-68.6 ^a	-33.8
	I	-72	-73 ^a	-59	-33
Average Range..		-59	-69	-63	-33
		20	9	10	3
Motor.....	C	-58.5	-62.4	-67	-34
	D	-62	-74	-68	-40
	I	-63	-63	-63 ^a	-34
	K	-54	-57	-66	-35
Average Range..		-59	-64	-66	-36
		9	17	5	6
Method D 736...	E	<-61	<-61	-49	-56.5
	I	-59	<-62	-45-48 ^a	-49
	K	<-62	<-62	<-62	<-62

^a Estimated from report of laboratory.

temperature, this method provided no data on T_b .

Test Program

Five materials listed in Table I were used in this study. The first four of these were the same compounds as those used in the earlier studies of other properties. Seven laboratories participated, using the test methods listed in Table II. In Method D 746, one to five specimens $\frac{1}{4}$ in. wide were tested simultaneously. The brittleness tem-

peratures were calculated both by formula and graphically. Method D 736 was used as written except that ten specimens were tested at each temperature and laboratories were requested to include the lowest temperature at which all specimens did not fail, the highest temperature at which all failed, and at least one temperature between these extremes. This was done to compare these tests, if possible, on the same basis.

The results of this study are tabulated in Table III, and are shown in Fig. 2. From the data obtained, it can be seen

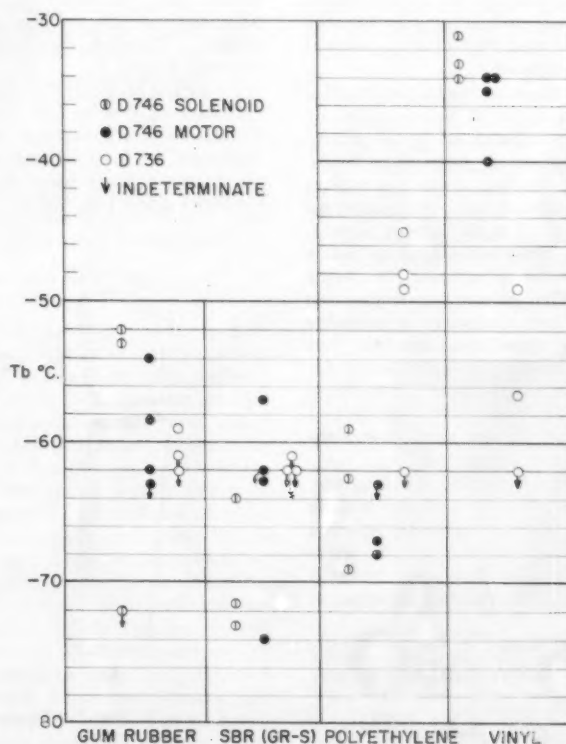


Fig. 2.—Results of first test program.

that T_b for some of the tests on gum rubber, SBR, and polyethylene was indeterminate, either because of inability of the laboratory to obtain a sufficiently low temperature or because some specimens did not break into pieces as originally specified for failure condition. More than half of the data from Method D 736 were indeterminate. It was concluded, therefore, that achievement of the criterion of "complete

separation" specified in Method D 746-52 T is unreasonable for some materials, such as gum rubber, and that "any visible crack" would be a more meaningful criterion. It was decided to repeat the measurements by Method D 746, using the new criterion in order to obtain a better measure of the reproducibility among laboratories. It was also decided that a neoprene sample should be tested along with the gum

rubber and SBR (GR-S). This neoprene compound had been used in earlier studies by ASTM Committee D-20 on Plastics and was known to have a very sharply defined brittleness temperature. Six laboratories participated in this second study. The results are summarized in Table IV and are shown in Fig. 3. In this summary, figures in parentheses are those determined by the graphical method while the other figures were determined by calculation from the formula. The advantage and validity of the "any visible failure" criterion are obvious from the fact that the T_b is now determinate in practically all cases. The improved reproducibility among laboratories using different types of equipment is indicated by the greater precision of the data.

Discussion

Since the scope of the work of the task group was to examine existing methods, no research or development work was undertaken to determine causes of poor reproducibility or to improve the method of test. It is clear that more work on the present methods would be useful. Considerable study has gone into the present version of Method D 746-55 T and, with minor editorial changes, it will soon be considered for adoption as standard.

Although the speed of impact, one of the most important variables of the test, is specified with reasonable tolerance, it is a difficult parameter to measure. Improved methods developed recently for measuring the velocity of the striker have shown that some machines do not operate at the specified velocity, (13,14,15,16). These techniques should lead to better control of velocity and consequently better reproducibility.

It is re-emphasized that brittleness temperature is not a fundamental property of a material. Any measure used is a function of many variables, particularly mode and rate of straining. Materials which elongate more than 500 per cent at moderate speeds may exhibit signs of brittle fracture at very high speeds, even at room temperatures. The fact that a material has a brittleness temperature of -40°C does not necessarily mean that it will not exhibit brittle failures above that temperature in any application nor, conversely, that it will always fail in all applications below -40°C . The so-called "brittleness temperature" is an empirical value useful for specification and identification of materials, but as stated in the scope of Method D 746, it "does not necessarily measure the lowest temperature at which the material may be used."

TABLE IV.—INTERLABORATORY STUDY OF ASTM METHOD D 746, BRITTLENESS TEMPERATURE IN DEG CENT.

	Neoprene		Gum Rubber		SBR (GR-S)	
	(1)	(2)	(1)	(2)	(1)	(2)
American Cyanamid Co. (Solenoid).....	-39	-39	< -55	-54	-74 ^b	-67
	(-39)	(-39)		(-53)	(-73)	(-68)
The Bakelite Co. (Motor)...	-38	-	-	-61.5	-63	-
Du Pont Polychemicals (Motor).....	-41	-40	< -70	-57	-	-
	(-41)	(-40)		(-57)	(-72)	(-70)
B. F. Goodrich Co. (Solenoid).....	-39	-37	< -74	-53	-73 ^b	-66
	(-39)	(-38)	(-74)	(-52)	(-73)	(-66)
Rock Island Arsenal (Solenoid).....	-38	> -33	< -74	> -69	-67	-58
U. S. Rubber (Motor).....	-38	-34	< -66	-55	-66 ^b	-57
	(-38)	(-34)	(-66)	(-56)	(-66)	(-59)

(1) T_b for complete breaks.

(2) T_b for any crack.

^a Figures in parentheses determined by graphical method.

^b One point estimated for calculation.

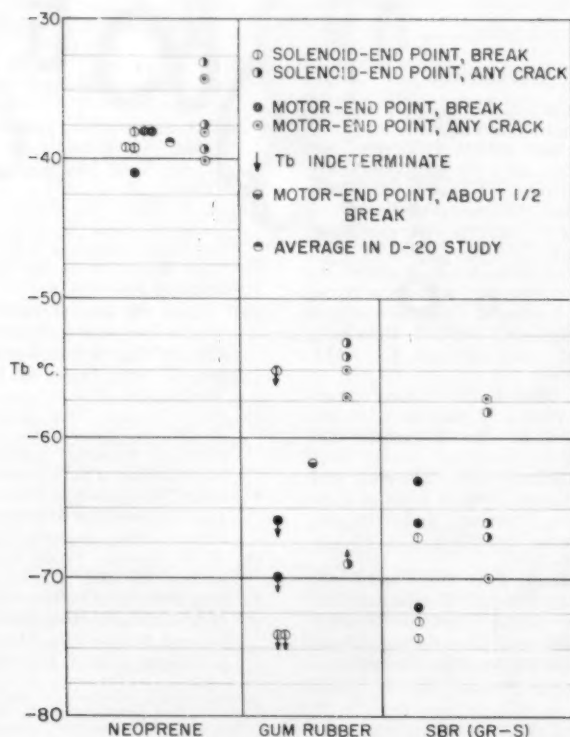




Fig. 3.—Results of second test, using Method D 746 only.

TABLE V.—SIGNIFICANT FACTORS IN BRITTLINESS TESTING AT LOW TEMPERATURES.

Method	Instrument	Specimen	Cond. Time	Force Applied	Rate	Number Tested	Report
Method D 736 - 54 T...	Hand operated	D 412 Die C 0.080 in. ± 0.010 in.	5 hr (air)		"rapidly as possible"	2 per temperature (minimum)	pass or fail at test temperature
Method D 746 - 55 T...	Motor driven	1.00 ± 0.25 in. long 0.075 ± 0.010 in. thick 0.25 ± 0.02 in. wide	3 ± 0.5 min (liquid)		6.0 - 7.0 ft per sec	10 per temperature (minimum)	(a) T _b = temperature for 50 per cent failure (b) Per cent failing at test temperature
	Solenoid actuated	T-50 Test specimens of Method D 599 and modified T-50 specimens have been used					

Conclusions

In addition to providing brittleness temperature data on the four materials for the correlation study, this work has led the task group to the following conclusions:

1. Method ASTM D 746 is presently the only standard method suitable for routine determination of low-temperature brittleness.

2. The failure criterion now specified in Method D 746 is preferable to the former specification.

3. There is no significant difference between results from the motor and solenoid operated equipment provided each is used as specified.

4. Our data support the conclusion of Committee D-20 that there is normally no significant difference between results obtained by calculation and by the graphical method.

5. Further refinement of speed control and possibly the addition of speed indication should improve the reproducibility of this method.

Acknowledgment:

The Task Group acknowledges the contribution of the men and organizations whose efforts, in the usual cooperative spirit of ASTM, have made this work possible: B. G. Labbe formerly of the Government Laboratories, University of Akron, now of Firestone Tire and Rubber Co.; R. H. Carey, The Bakelite Co.; and J. B. DeCoste of the Bell Telephone Laboratories provided the test materials.

These men and F. L. Graves of the American Cyanamid Co., L. V. Cooper, Firestone Tire and Rubber Co.; Ross Shearer, B. F. Goodrich Research Center; A. C. Hanson, Rock Island Arsenal;

W. E. Scoville, Jr., U. S. Rubber Co.; and the Du Pont Polychemicals Dept. obtained the test data.

L. Boor of the U. S. Army Quartermaster Corps, R. E. Ofner of the Rock Island Arsenal and F. S. Conant of Firestone Tire and Rubber Co. assisted as members of the task group in the planning and summation of the work under the chairmanship of F. M. Gavan of the Armstrong Cork Co. The study was made under the auspices of Subcommittee 27 of Committee E-1, whose chairman, R. S. Havenhill, of St. Joseph Lead Co., has given the group constant support and encouragement.

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Mechanical and Physical Characteristics of Jewelry Bronze, 87.5 per cent Strip

By DELMAR E. TROUT

THERE are no ASTM specifications which include Jewelry Bronze, 87.5 per cent for any basic mill form. However, for many years this alloy has been produced and consumed in volume quantities, primarily in wire form. Because of its almost natural gold color, it has found use as a base for gold plate and as a substitute for gold in costume jewelry items. Perhaps its greatest application has been in wire form for slide fastener component parts. In the past few years there has been an increasing interest and use of this alloy in strip form for such applications as eyelets, compacts, emblems, etched articles, lipstick containers, and plaques. The alloy has excellent cold-working and good hot-forming characteristics and can be processed by all common fabrication processes.

Reliable mechanical property data for strip has been lacking, and experience has shown that an interpolation of Commercial Bronze, 90 per cent (ASTM Specification B 36, alloy No. 2), and Red Brass, 85 per cent (ASTM Specification B 36, alloy No. 3) requirements did not warrant sufficient assurance that the resultant properties for Jewelry Bronze, 87.5 per cent were either correct or satisfactory for specification purposes. In November 1954, the Copper and Brass Research Assn. (CABRA) approved this alloy as a standard and issued Alloy Data Sheet 7.1. Some of the properties in this data sheet appeared to contain interpolated values for flat products due to the lack of more complete data.

It was therefore deemed desirable to determine the mechanical properties for this alloy for both rolled and annealed tempers. This information could then serve for both internal control and specification purposes. Also, some of the more widely used physical properties were determined.

Preparation of Material

All test specimens were prepared from a single 3000-lb cast bar with an as-cast cross-section of $3\frac{1}{2}$ by $25\frac{1}{2}$ in.

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¹ Specification for Brass Plate, Sheet, Strip, and Rolled Bar (B 36—56), 1956 Supplement to Book of ASTM Standards, Part 2, p. 18.

There are no ASTM specifications for this 87.5 per cent copper, 12.5 per cent zinc alloy in basic mill form

This bar had the following chemical composition:

Copper.....	87.29 per cent
Lead.....	0.01 per cent
Iron.....	0.02 per cent
Tin.....	0.01 per cent
Zinc.....	Remainder

This bar was processed to 0.500 in. according to the following mill procedure:

1. Corner mill;
2. Roll to 1.255 in. thick in five passes;
3. Anneal at 750 C;
4. Roll to 0.535 in. thick in 3 passes;
5. Crop ends;
6. Anneal at 750 C; and
7. Flatten and overhaul to 0.500 in. thick.

Sufficient material was selected to yield 18 coils, each of approximately 60 lb of 5 in. width by 0.040 in. gage and finished in various tempers:

Descriptions of the 18 coils follow:

Coil	Finished Temper
No. 1...	0.025 mm nominal annealed
No. 2...	0.050 mm nominal annealed
No. 3...	Quarter-hard from 0.025 mm
No. 4...	Quarter-hard from 0.050 mm
No. 5...	Half-hard from 0.025 mm
No. 6...	Half-hard from 0.050 mm
No. 7...	Three-quarter-hard from 0.025 mm
No. 8...	Three-quarter-hard from 0.050 mm
No. 9...	Hard from 0.025 mm
No. 10...	Hard from 0.050 mm
No. 11...	Extra hard from 0.025 mm
No. 12...	Extra hard from 0.050 mm
No. 13...	Spring from 0.025 mm
No. 14...	Spring from 0.050 mm
No. 15...	Extra spring from 0.025 mm
No. 16...	Extra spring from 0.050 mm
No. 17...	12 Brown and Sharpe numbers from 0.025 mm
No. 18...	12 Brown and Sharpe numbers from 0.050 mm

The reduction prior to the ready-to-finish anneal was extra hard temper (6 B & S Nos.) for all cases. Ready-to-finish gages were accurately checked, as were the finished gages, to permit calculation of exact percentage reductions for eventual use in plotting the derived test data. All coils were checked for grain size at each annealing point, and in all cases the average diameter was within 0.005 mm of that desired at the nominal annealed temper.

Samples were selected from coil Nos. 5, 9, 11 and 13, and were subsequently annealed in a Lindberg laboratory fur-

nace at appropriate temperature increments each 1 hr at temperature in order to determine the annealing characteristics.

Properties Investigated

The following tests were performed on the rolled-and-annealed temper specimens where applicable:

1. Tensile strength, psi,
2. Yield strength (0.5 per cent extension under load), psi,
3. Yield strength (0.2 per cent offset), psi,
4. Yield strength (0.1 per cent offset), psi,
5. Elastic limit, psi,
6. Proportional limit, psi,
7. Elongation in 2 in., per cent,
8. Shear strength, psi,
9. Rockwell hardness, B scale,
10. Rockwell hardness, F scale,
11. Rockwell hardness, 30T scale,
12. Rockwell hardness, 15T scale,
13. Knoop hardness (KHN) (1000-g load),
14. Microstructure and grain size, mm,
15. Modulus of elasticity, psi,
16. Electrical resistivity, and
17. Electrical conductivity.

Methods of Test

Tensile properties were determined in accordance with the methods of test prescribed in the ASTM Methods of Tension Testing of Metallic Materials



DELMAR E. TROUT, supervisor of metallurgical research, Scovill Manufacturing Co., Waterbury, Conn. is responsible for the development of new alloys, new or improved metallurgical methods and properties, and improvement of present alloys and their properties and characteristics. For the past several years Mr. Trout has been active in the work of ASTM Committee B-5 on Copper and Copper Alloys.

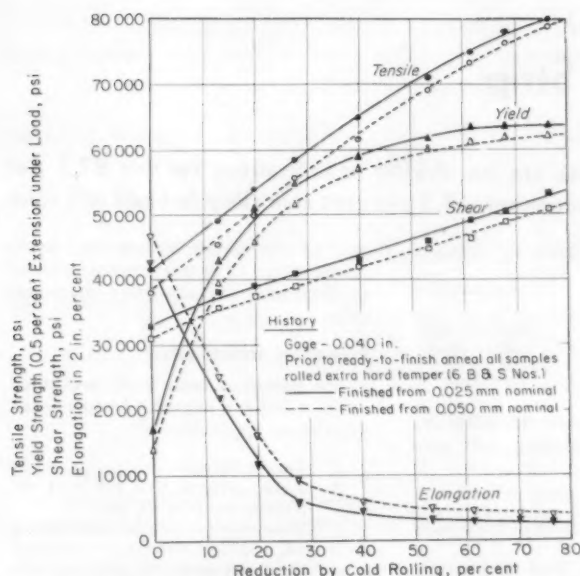


Fig. 1.—Effect of cold rolling on mechanical properties of jewelry bronze, 87.5 per cent strip.

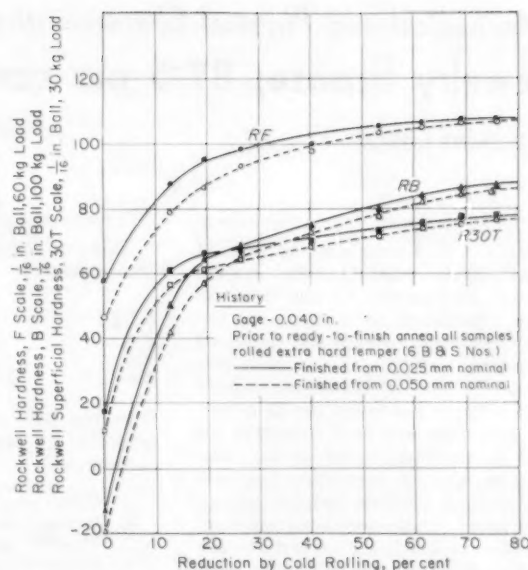


Fig. 2.—Effect of cold rolling on hardness of jewelry bronze, 87.5 per cent strip.

(E S)² using test specimens conforming to Fig. 6 of those methods. All tension specimens were prepared by blanking and shaving in a double-die tool in a 50-ton single action press. The specimens were tested on a 60,000-lb Baldwin-Tate-Emery universal testing machine.

The yield strength at 0.5 per cent extension under load was determined using a Riehle Clamp-O-Matic extensometer.

The proportional limit, elastic limit, yield strength at both 0.1 and 0.2 per cent offset and modulus of elasticity were determined, using a Baldwin-Southwark automatic stress-strain recorder with a 2-in. gage length extensometer integrated in the testing machine.

Because of the character of the stress-strain curve for copper-zinc alloys, which do not exhibit a definable yield point, it is difficult to determine the stress at which inelastic action begins; thus the elastic limit and the proportional limit (these terms frequently being used interchangeably) are most difficult to evaluate. By definition the elastic limit is the greatest stress which a material is capable of sustaining, without any permanent strain remaining, upon complete release of the stress;

whereas the proportional limit is the greatest stress which a material is capable of sustaining without any deviation from proportionality of stress-to-strain (Hooke's law).

For our tests, the elastic limit was deemed to be the maximum stress at which, upon release of the load, no measurable permanent set was noted. The proportional limit was, as noted above, taken to be the greatest stress on the Hooke's law portion of the stress-strain curve.

Elongation was determined by matching the ends of the fractured tension specimen and measuring the change in length of the previously scribed 2-in. gage marks.

Shear strength was determined using a specially designed jig which contained a disk $2\frac{1}{4}$ in. diameter of the material to be tested, and from which is blanked a disk of $1\frac{1}{2}$ in. diameter using a constant hold-down pressure on the larger disk. The force required to blank the $1\frac{1}{2}$ -in. disk, when related to the sheared area, gives the shear strength. This test was performed under compression on the same testing machine as was used for the tension tests.

Rockwell hardness values of both ends of the tension specimens were determined prior to testing in accordance with ASTM Tentative Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (E 18).³

Knoop hardness numbers (KHN) were determined on a Tukon microhardness tester using a 1000-g load. Small specimens were cut from the tension

specimen skeleton scrap and the surface to be tested was hand polished through 0000 emery paper.

The microstructure and grain size studies were made on samples prepared in accordance with the methods of test prescribed in ASTM Methods E 3⁴ and E 79.⁵ A modified chromic acid etching reagent was used for studies of both the microstructure and grain size. The grain size was estimated using a GSL-Micrographex Comparator.

Electrical properties were calculated from resistance measurements made on 0.5 in. by 0.040 in. by 1 m slit samples using the Kelvin bridge method. All values were corrected to 20 C.

Test Results

All tests were run in duplicate and generally the results were within experimental error. If necessary, additional samples were tested. All plotted data points represent averages of the individual test results.

Figures 1 and 2 are standard-form cold-rolling curves, and their over-all pattern is typical for copper-zinc alloys. An interesting fact to note is the difference in work-hardening rates of the material in tension as compared to that in shear (Fig. 1). Whereas the tensile strength for 80 per cent reduction by cold rolling is double that of the annealed temper, there is but a two-thirds increase in shear strength for comparable reduction. This has been found to be a general characteristic of most copper alloys. The flattening of the hardness curves (Fig. 2) after about 20 per cent reduction by cold rolling em-

² (E 8 - 54 T), 1955 Book of ASTM Standards, Part 1, p. 1551; Part 2, p. 1246.

³ (E 18 - 55 T), 1955 Book of ASTM Standards, Part 1, p. 1590; Part 2, p. 1285.

⁴ Tentative Methods of Preparation of Metallographic Specimens (E 3 - 46 T), 1955 Book of ASTM Standards, Part 2, p. 1149.

⁵ Tentative Methods for Estimating the Average Grain Size of Wrought Copper and Copper-Base Alloys (E 79 - 49 T), 1955, Book of ASTM Standards, Part 2, p. 1345.

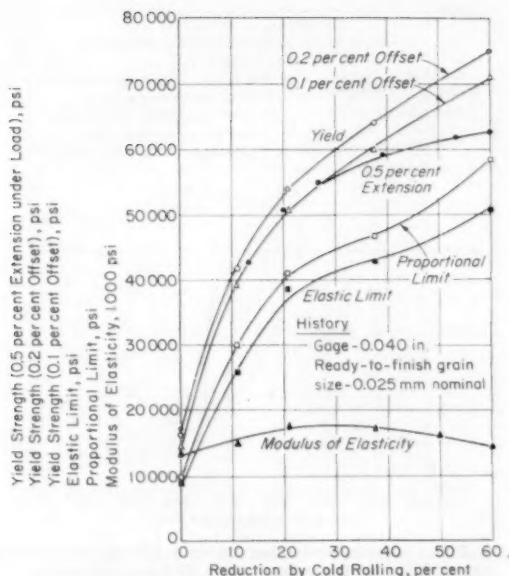


Fig. 3.—Effect of cold rolling on mechanical properties of jewelry bronze, 87.5 per cent strip.

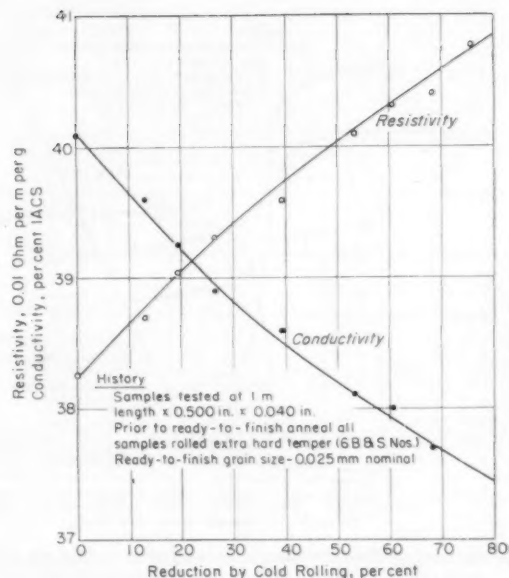


Fig. 4.—Effect of cold rolling on electrical properties of jewelry bronze, 87.5 per cent strip.

phasizes the difficulties or unreliability inherent in the use of hardness tests for specification purposes.

The copper alloy industry has adopted the extension method as a standard method of testing for yield strength. The advantage of this method is that it can be determined directly whereas the offset method requires a time-consuming stress-strain curve. As long as the yield strength is defined and the method is standard within an industry, its usefulness as such is obvious. Because copper alloys have similar stress-strain curves, the 0.5 per cent extension method is entirely reliable and reproducible from sample to sample. As can be noted from Fig. 3, the yield strength by both the extension and offset methods is similar for annealed tempers and has similar slopes and values up to about 30 per cent reduction by cold rolling. However, above 30 per cent the extension yield strength changes slope and increases at a lesser rate for increased rolling reductions. Comparison of the extension method yield strength and the elongation curves of Fig. 1 shows them to appear inversely proportional. Extension yield strength

is more directly related to ductility than is offset yield strength. Offset yield strength is desirable for a few engineering applications; therefore its determination was felt necessary to provide complete data on this alloy.

It would be expected that elastic and proportional limits should be very close in value. However, when tested according to the basic definitions, the proportional limit is higher than the elastic limit by 1000 to 8000 psi. Refinement of test methods and minimization of all test errors should cause these properties to approach one another.

The character of the modulus of elasticity curve in Fig. 3 warrants comment. The modulus of elasticity is usually published as a constant value regardless of temper. However, it can be seen that this property is maximum at about 30 per cent reduction by cold rolling, and the values for the annealed and 60 per cent reduction tempers are similar. For some engineering calculations, this change in modulus should be taken into consideration. It is interesting to note that the character of the modulus of elasticity curve shown in Fig. 3 is similar to many shown by Gohn, Guerard, and Freynick.⁶

Figure 4 illustrates the effect of cold rolling on the electrical properties of Jewelry Bronze, 87.5 per cent. With increasing amounts of cold rolling there is a proportional decrease in conductivity, with a similar increase in resistivity. This effect is normal for copper-zinc alloys.

Figures 5 and 6 illustrate the effect

of the annealing temperature on the mechanical properties and the grain size of Jewelry Bronze, 87.5 per cent. The ordinate scale for grain size is a pseudo-logarithmic scale. A linear scale for grain size *versus* a linear scale for temperature results in a hyperbolic type of curve whereas this pseudo-logarithmic scale results in a linear relationship of grain size *versus* temperature. Another advantage of this scale is that the ASTM annealed temper ranges will have similar linear displacements. For example, the ordinate length 0.015 to 0.035 mm is approximately the same as 0.035 to 0.070 mm. From Figs. 5 and 6, the recrystallization and softening temperatures can be readily selected and the values and rates of change of various properties during annealing can be determined.

It is interesting to note that the rate of change of mechanical properties for the annealed tempers is very small over a very wide spread in grain size; wide variations in grain size have little effect on mechanical properties. Grain size determinations closer than the ASTM rounding-off procedure (E 29)⁷ have little significance in relation to mechanical properties.

A maximum is noted in the elongation curve (Fig. 5) at a grain size of about 0.035 mm. Above 0.035 mm the elongation decreases slightly indicating a loss of ductility at larger grain sizes. Several years ago we attempted to evaluate the Olsen ductility cup test and found a similar condition for copper-zinc alloys containing 85 per cent

⁶ G. R. Gohn, J. P. Guerard, and H. S. Freynick, "The Mechanical Properties of Wrought Phosphor Bronze Alloys," Am. Soc. Testing Mats. (1955). (Issued as Separate Publication ASTM STP No. 133.)

⁷ Recommended Practices for Designating Significant Places in Specified Limiting Values (E 29 - 50), 1955 Book of ASTM Standards, Part 1, p. 1634; Part 2, p. 133; Part 3, p. 1940; Part 4, p. 1318; Part 5, p. 1416; Part 6, p. 1662; Part 7, p. 1605; 1956 Chemical Analysis of Metals, p. 52.

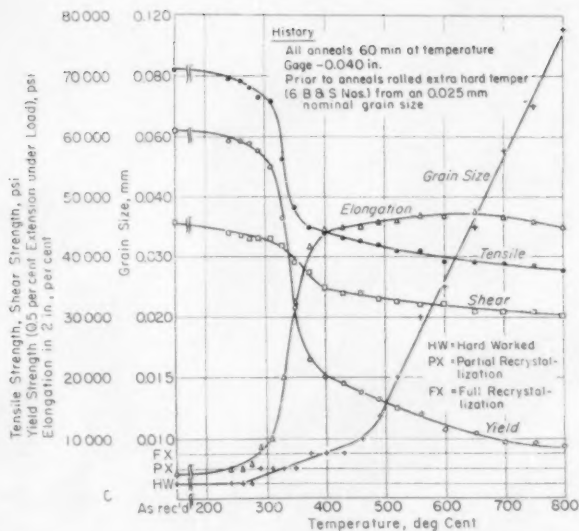


Fig. 5.—Annealing characteristics of jewelry bronze, 87.5 per cent strip.

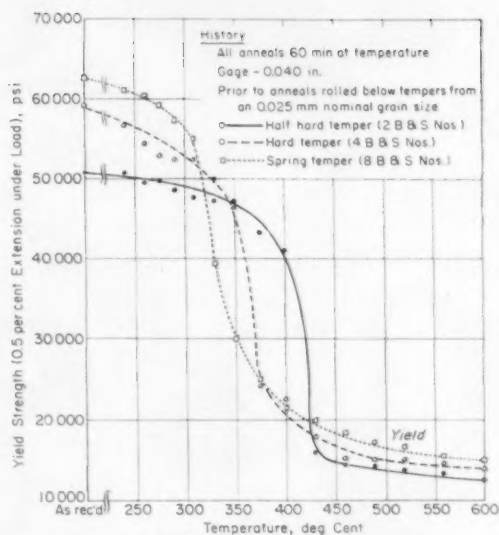


Fig. 7.—Effect of prior reduction by cold rolling on annealing characteristics of jewelry bronze, 87.5 per cent strip.

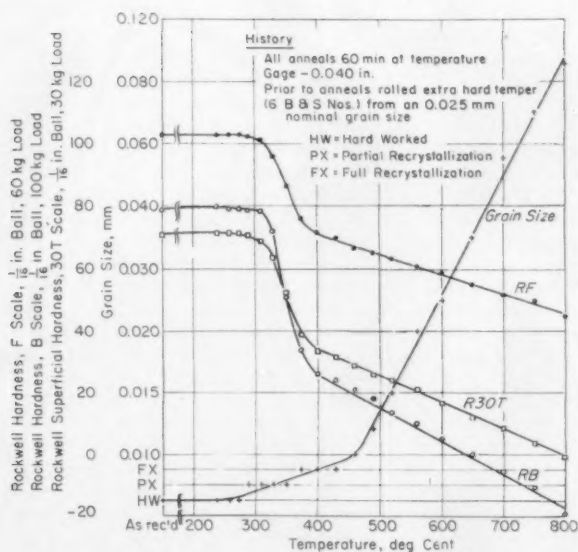


Fig. 6.—Annealing characteristics of jewelry bronze, 87.5 per cent strip.

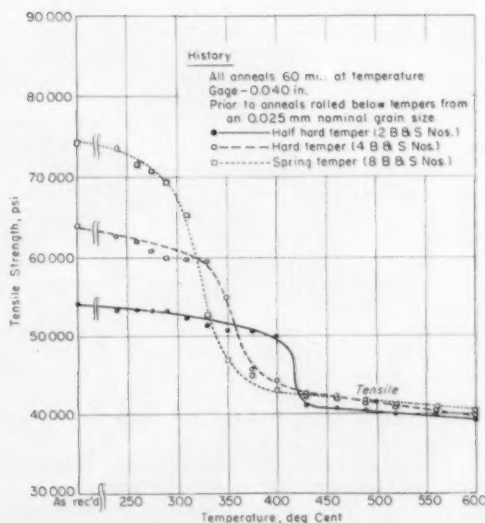


Fig. 8.—Effect of prior reduction by cold rolling on annealing characteristics of jewelry bronze, 87.5 per cent strip.

or more copper. For these red alloys the maximum cup height or ductility was at a grain size of 0.035 mm, whereas for Cartridge Brass, 70 per cent, the maximum ductility was at a grain size

of 0.070 mm.

Data and information on the effect of prior reduction by cold rolling on mechanical properties (Figs. 7 to 9) and grain size (Fig. 10) are most valu-

able both to the producer and the fabricator. Study of these figures as well as of Figs. 5 and 6 reveals that a minimum reduction of extra hard (6 B & S Nos.) or 50 per cent by cold rolling was

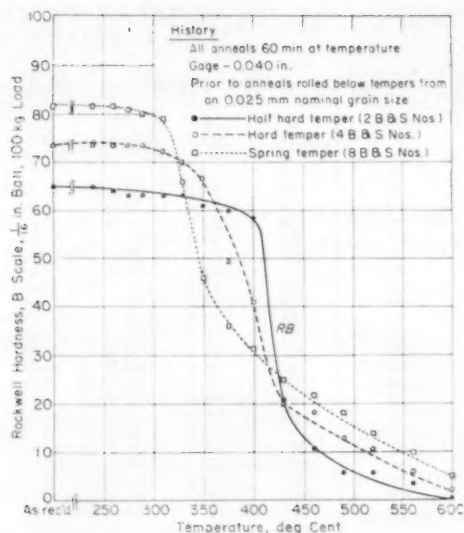


Fig. 9.—Effect of prior reduction by cold rolling on annealing characteristics of jewelry bronze, 87.5 per cent strip.

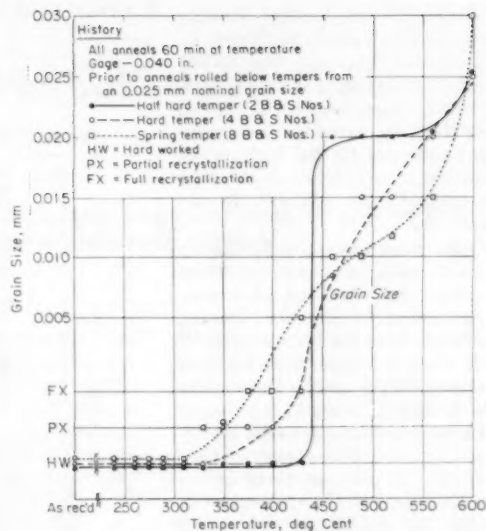


Fig. 10.—Effect of prior reduction by cold rolling on annealing characteristics of jewelry bronze, 87.5 per cent strip.

necessary between annealings to result in normal and uniform annealed properties and characteristics. The softening temperatures for various prior reductions defined as the midpoint between initial recrystallization and an 0.010 mm grain size are as follows:

Prior Reduction Temper	Softening Temperature, deg Cent (1 hr at temperature)
Half-hard, 2 B & S Nos.	425
Hard, 4 B & S Nos.	370
Extra Hard, 6 B & S Nos.	330
Spring, 8 B & S Nos.	330

The recrystallized grain size for half-hard temper (2 B & S Nos.) was 0.020 mm as compared to a nominal ready-to-finish grain size of 0.025 mm. All other tempers showed fine recrystallized grain sizes (0.005 to 0.006 mm) with increasing uniformity in direct relationship to the prior reduction.

Many times it is required that a complicated drawn article be finish-annealed with an over-all hardness or grain size within a certain range. Actual metal thickness reductions on the final operations may vary from 2 to 5 per cent through an intermediate reduction to perhaps 40 to 45 per cent on other sections of the part. As can be seen from Figs. 7 to 10 to attain close hardness or grain size control is difficult. Also, in complicated drawn shells it is possible to anneal the heavily

* Values for alloys other than Jewelry Bronze, 87.5 per cent taken from original Scoville data and from CABRA alloy data sheets 5, 6, and 7.

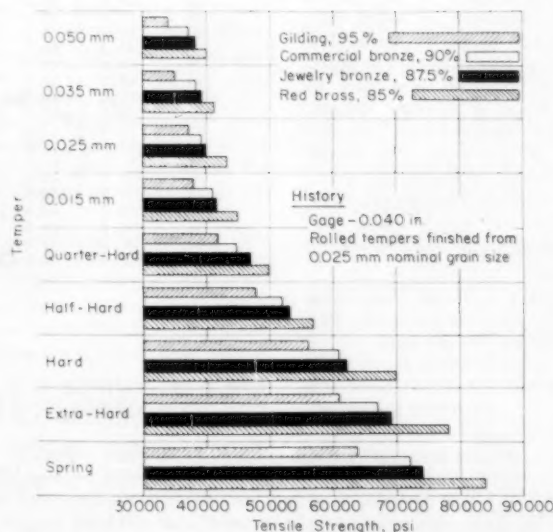


Fig. 11.—Comparative tensile strengths of some copper-zinc alloys.

reduced portion without softening the area of lower reduction.

Figure 11⁸ shows the comparative tensile strength at various tempers of Gilding, 95 per cent; Commercial Bronze, 90 per cent; Red Brass, 85 per cent (ASTM B 36 Alloys No. 1, 2, and 3 respectively); and Jewelry Bronze, 87.5 per cent. As revealed in this chart, the properties of Jewelry Bronze, 87.5 per cent, are very similar

to those of Commercial Bronze, 90 per cent, especially for rolled tempers. Therefore an interpolation of the properties of the 90 per cent and 85 per cent copper-zinc alloys is incorrect and substantiates our original objective for this investigation.

Sufficient data had been collected from tests on both breakdown and the finished tempers, together with earlier data, to compile a hardness conversion

chart applicable to Jewelry Bronze, 87.5 per cent flat products (Table I). Comparing this chart with the ASTM Hardness Conversion Tables for Cartridge Brass, 70 per cent (E 33)¹ it can be seen that the cartridge brass conversion hardnesses for the high-copper alloys can be in error by as much as 3.5 points between the Rockwell B and F scales.

The conversion values for the Knoop hardness number are not consistent with those for Rockwell hardness. The conversion of Jewelry Bronze, 87.5 per cent, from the B scale to the F or 30 T scale is higher than for Cartridge Brass, 70 per cent. The conversion of Jewelry Bronze, 87.5 per cent from the Rockwell B scale to the Knoop hardness number is lower than for Cartridge Brass, 70 per cent, based on the Wilson Mechanical Instrument Co. Chart No. 52. Additional work would be necessary to resolve or explain this discrepancy.

Conclusions

The mechanical properties of Jewelry Bronze, 87.5 per cent more nearly approximate those of Commercial Bronze, 90 per cent than those of Red Brass, 85 per cent, especially for rolled tempers. For usage based on mechanical properties alone there appears to be no significant or improved properties to warrant a choice of Jewelry Bronze, 87.5 per cent in preference to Commercial Bronze, 90 per cent. The choice of Jewelry Bronze, 87.5 per cent would be generally dependent upon a property, such as color, electrical properties, thermal characteristics, color being the most important.

Our experience indicates that Table I is reasonably accurate for copper-zinc alloys with 82 to 100 per cent copper and that it is sufficiently accurate to serve as a basis for the development of a high-copper alloy standard conversion chart.

For our own use we prepared Table II which contains tensile strength requirements for rolled tempers and approximate Rockwell hardness values for both rolled and annealed tempers, patterned after the tabulated form for requirements in ASTM Specification B 36. The data in Table II have been used for our internal control and specification purposes for Jewelry Bronze, 87.5 per cent flat products for almost one year, with no serious discrepancies or inconsistencies noted. These tabulated data may prove valuable for specification work for this alloy, especially by ASTM Committee B-5 on Copper and Copper Alloys.

¹ (E 33-42), 1955 Book of ASTM Standards, Part 2, p. 1318.

TABLE I.—HARDNESS CONVERSION CHART FOR JEWELRY BRONZE, 87.5 PER CENT FLAT PRODUCTS.

Rockwell Hardness			Knoop Hardness Number, over 500 g	Rockwell Hardness			Knoop Hardness Number, over 500 g
B Scale	F Scale	30T Scale		B Scale	F Scale	30T Scale	
0...	60.5	20.5	59	51	88.0	53.5	95
1...	61.0	21.0	59	52	88.5	54.0	96
2...	61.5	21.5	60	53	89.0	54.5	97
3...	62.0	22.0	60	54	89.5	55.5	98
4...	62.5	23.0	61	55	90.0	56.0	99
5...	63.0	23.5	61	56	90.5	56.5	100
6...	64.0	24.0	62	57	91.0	57.0	101
7...	64.5	25.0	62	58	92.0	58.0	103
8...	65.0	25.5	63	59	92.5	58.5	104
9...	65.5	26.0	63	60	93.0	59.0	105
10...	66.0	27.0	64	61	93.5	59.5	106
11...	66.5	27.5	65	62	94.0	60.5	108
12...	67.0	28.0	65	63	94.5	61.0	109
13...	67.5	28.5	66	64	95.0	61.5	110
14...	68.0	29.5	66	65	95.5	62.5	112
15...	68.5	30.0	67	66	96.0	63.0	113
16...	69.0	31.0	68	67	96.5	63.5	115
17...	69.5	31.5	68	68	97.0	64.0	116
18...	70.0	32.0	69	69	97.5	65.0	118
19...	70.5	33.0	70	70	98.0	65.5	119
20...	71.0	33.5	71	71	98.5	66.0	121
21...	72.0	34.0	72	72	99.0	67.0	122
22...	72.5	34.5	72	73	100.0	67.5	125
23...	73.0	35.5	73	74	100.5	68.0	126
24...	73.5	36.0	74	75	101.0	68.5	128
25...	74.0	36.5	74	76	101.5	69.5	130
26...	74.5	37.0	75	77	102.0	70.0	132
27...	75.0	38.0	75	78	102.5	70.5	134
28...	75.5	38.5	76	79	103.0	71.5	136
29...	76.0	39.0	77	80	103.5	72.0	138
30...	76.5	39.5	78	81	104.0	72.5	140
31...	77.0	40.5	78	82	104.5	73.5	142
32...	78.0	41.0	79	83	105.5	74.0	144
33...	78.5	41.5	80	84	106.0	74.5	146
34...	79.0	42.5	81	85	106.5	75.0	149
35...	79.5	43.0	82	86	107.0	76.0	151
36...	80.0	43.5	82	87	107.5	76.5	153
37...	80.5	44.5	83	88	108.0	77.0	155
38...	81.0	45.0	84	89	108.5	78.0	158
39...	81.5	45.5	85	90	109.0	78.5	160
40...	82.0	46.5	86	91	109.5	79.0	162
41...	82.5	47.0	87	92	110.0	80.0	164
42...	83.0	47.5	87	93	...	80.5	167
43...	83.5	48.0	88	94	...	81.0	170
44...	84.0	49.0	89	95	...	81.5	...
45...	85.0	49.5	90	96	...	82.5	...
46...	85.5	50.0	91	97	...	83.0	...
47...	86.0	50.5	92	98	...	83.5	...
48...	86.5	51.5	93	99	...	84.5	...
49...	87.0	52.9	94	100	...	85.0	...
50...	87.5	52.5	94				

TABLE II.—TENSILE STRENGTH REQUIREMENTS FOR ROLLED TEMPERS AND APPROXIMATE ROCKWELL HARDNESS VALUES FOR BOTH ROLLED AND ANNEALED TEMPERS FOR JEWELRY BRONZE, 87.5 PER CENT FLAT PRODUCTS.

Temper	Tensile Strength, psi		Approximate Rockwell Hardness*					
			B Scale		F Scale		30T Scale	
	Min	Max	Min	Max	Min	Max	Min	Max
Quarter-hard.....	42 000	52 000	29	58	71	89	39	58
Half-hard.....	48 000	58 000	52	68	89	98	54	64
Three-quarter-hard.....	53 000	63 000	61	73	59	68
Hard.....	58 000	67 000	67	77	64	70
Extra hard.....	65 000	73 000	74	81	68	73
Spring.....	70 000	78 000	78	83	71	74
Extra spring.....	74 000	82 000	81	86	73	76
0.050 mm.....	48	58	6	18
0.035 mm.....	52	62	10	23
0.025 mm.....	55	67	14	29
0.015 mm.....	58	76	18	40

* For rolled tempers B and F scales apply to metal 0.020 in. thick and over, for 30T scale 0.012 in. thick and over. For annealed tempers F scale applies to metal 0.020 in. thick and over, for 30T scale 0.015 in. thick and over.

Four Methods of Determination of Carbon Dioxide in Solid Fuels

By PETER O. KRUMIN and KARL SVANKS

A comparative study of apparatus and procedures for gravimetric, titro-metric, gas-volumetric, and pressometric methods

Significance of Tests

IF SOLID fuel contains carbonates, the volatile matter determined according to ASTM Method D271-48¹ includes carbon dioxide evolved from carbonates, and the determined ash value is lower to that extent. The carbon determined in ultimate analysis represents total carbon in coal and in mineral carbonates. Therefore, an accurate determination of total carbon dioxide in mineral solid fuels is of significance for use in the correction of analytical results, in particular for correction of the volatile matter and organic values, and for calculation of coal analyses to a mineral matter-free basis.

Small amounts of carbonates, combined principally with calcium, magnesium, and iron, in compounds such as: calcite (CaCO_3), dolomite ($\text{CaCO}_3 \cdot \text{MgCO}_3$), ankerite or brass-stone ($2\text{CaCO}_3 \cdot \text{MgCO}_3 \cdot \text{FeCO}_3$), siderite (FeCO_3), witherite (BaCO_3), etc., occur in many coals as partings, cleats, inclusions, impregnations, nodules, or concretions. Comparatively large amounts of mineral carbonates occur in some coals. However, in many American oil shales the carbonate percentages are so high that volatile matter, fixed carbon, and ash values are of doubtful accuracy, if reported without corrections. For example, the carbonate CO_2 content in three Colorado oil shale samples used in this study was found to be 18.3 to 20.0 per cent by weight of the raw oil shale.

A negligible amount of CO_2 , referred to as "free carbon dioxide" is evolved when a coal sample is treated with boiling water. A further, and usually a larger quantity of CO_2 , referred to as "fixed carbon dioxide" is evolved if a coal sample after removal of free CO_2 is

treated with diluted mineral acid. The determination of CO_2 for use in the correction of analytical results should be concerned only with the total, that is with both free and fixed carbon dioxide (1).² Accordingly, the following study refers to the determination of total CO_2 .

General Considerations

Many methods have been proposed by various investigators for the determination of carbonate CO_2 , too numerous to be reviewed in a paper of this size. The methods proposed in recent years are essentially based upon chemical reactions and methods reported in analytical chemistry textbooks and other publications, the improvements being directed toward replacing earlier pieces of apparatus with those which are less fragile, more easily maintained and operated, and having increased accuracy achieved by excluding parts and procedures possessing potential sources of error. This statement applies also to the modifications of apparatus employed in this study.

The basic concepts underlying the determination of carbonate CO_2 are: a complete liberation of carbonate CO_2 from the sample under investigation and determination of the liberated CO_2 . Care must be taken that: (a) no CO_2 , except

that which is in the sample, is introduced in the apparatus by the air, distilled water, etc.; (b) no CO_2 is fixed in the apparatus except in the absorption vessel; and (c) all other compounds that would interfere with the determination of CO_2 are removed. In addition, by employing gravimetric methods, care should be taken that the absorbing medium must neither gain nor lose water.

Common Operations

The decomposition of carbonates present in the sample is common for different methods and generally is done by action of dilute mineral acid with 1 per cent wetting agent, preferably phosphoric acid (sp gr 1.3), or 5N hydrochloric acid. However, J. B. Nelson and D. E. Shipley (12) found that by employing phosphoric acid with coals which contained chalybite, ($\text{Fe,Mg})\text{CO}_3$, low and inconsistent results were sometimes obtained for the CO_2 content because of incomplete decomposition of chalybite by the phosphoric acid. For decomposition of the carbonate minerals, they suggested hydrochloric acid as the reagent which would deal adequately with all coals and cokes. Air-dried samples ground to pass a 250- μ (No. 60) sieve or, better, passing a 149- μ (No.

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KARL SVANKS, research associate, has been engaged in fuels analysis in the Engineering Experiment Station, The Ohio State University, since 1950. Formerly, he was an instructor in the Department of Chemical Engineering, UNRRA University in Munich, assistant in the Institute of Fuels Technology, Technical University in Vienna and instructor in the Department of Chemical Technology, University of Latvia.

NOTE.—DISCUSSION OF THIS PAPER IS INVITED, either for publication or for the attention of the author. Address all communications to ASTM Headquarters, 1916 Race St., Philadelphia 3, Pa.

¹ Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271-48), 1955 Book of ASTM Standards, Part 5, p. 949.

² The boldface numbers in parentheses refer to the list of references appended to this paper.

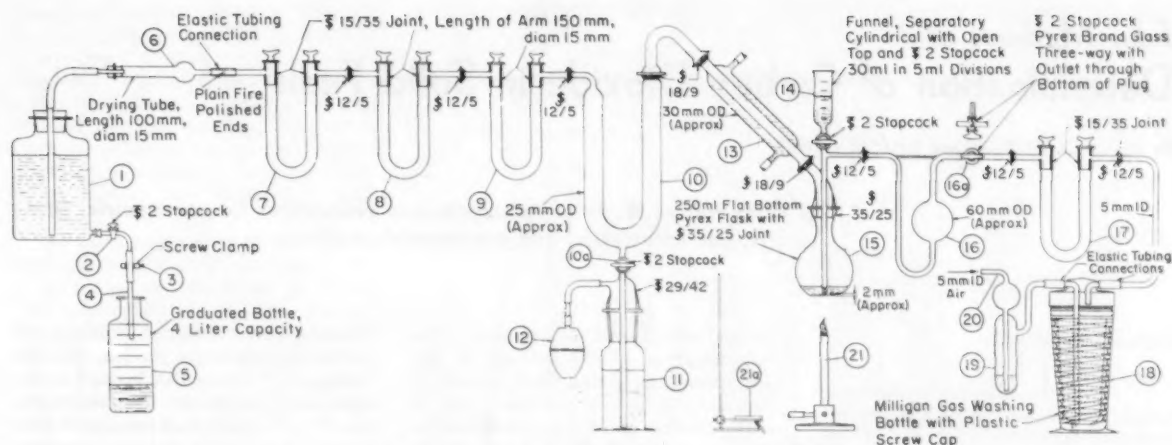


Fig. 1.—Diagram of apparatus for gravimetric determination of CO₂ consisting of:

- (1) 8-liter aspiration bottle with
- (2) stopcock on side tubulation.
- (3) Rubber connection tube with screw clamp.
- (4) Glass extension tube.
- (5) 4-liter graduate flask.
- (6) Drying tube, charged with soda asbestos (Ascarite) in the middle and anhydrous magnesium perchlorate (Dehydrite) on each end of the tube.
- (7) U-tube for CO₂ absorption, charged with soda asbestos (Ascarite) in the middle and anhydrous magnesium per-

- chlorate (Dehydrite) on each end of the tube.
- (8) U-tube charged with silver sulfate (or anhydrous copper sulfate) packed with layers of glass wool.
- (9) U-tube charged with granular magnesium perchlorate (Dehydrite).
- (10) U-shaped tube, packed with glass beads, and provided with drain tube and stopcock, 10a.
- (11) 250-ml cylinder charged with H₂SO₄ (sp gr 1.84).
- (12) Rubber bulb hand-pump.
- (13) Reflux condenser.
- (14) Tap funnel, charged with HCl (Approx. 5 N).
- (15) 250-ml reaction flask, pyrex glass.
- (16) Safety trap provided with 3-way stopcock, 16a.
- (17) U-tube charged with granular soda asbestos (Ascarite).
- (18) Milligan gas-washing bottle charged with H₂SO₄ (sp gr 1.84).
- (19) Air purifying bulb, of bubble counter or mercury check valve type, charged with H₂SO₄ (sp gr 1.84).
- (20) Air inlet tube.
- (21) Gas burner, or 21a electric heater.

100) sieve if much impurity is present, are used.

Scope

In an attempt to select the best known method or develop an improved, modified, or new method a comparative study of different methods has been made by the authors. The scope of this work includes study of apparatus and procedures for determination of CO₂ by the following methods: (a) gravimetric (b) volumetric, employing titration, (c) gas-volumetric, and (d) pressometric (or manometric).

Gravimetric Method (1, 3, 4, 7, 8)

The determination of CO₂ is made gravimetrically by complete decomposition of carbonates present in a weighed quantity of the sample under investigation by action of 5N hydrochloric acid with 1 per cent of wetting agent in a closed system, and by absorption of the liberated CO₂ after purification from interfering substances, in a weighed vessel containing a reagent for carbon dioxide absorption.

¹ In atmosphere of low humidity (60 per cent or lower) the U-tube by rubbing with dry cotton cloth will induce static charges. However, if a particle emitter is placed into the balance case, the troublesome static charges will be dissipated.

The apparatus shown in Fig. 1 has been found most satisfactory in conforming to the requirements outlined in the section on General Considerations and proved to be suitable for gravimetric determination of carbon dioxide in coal, coke, oil shale, and other materials with a wide range of carbonate content.

Procedure

When the apparatus is assembled as shown in Fig. 1, and made gas-tight by careful grinding, greasing, and tightening of all connections, the desired rate of air flow through the apparatus is established by regulating the screw clamp 3 (Fig. 1). Then the aspiration of air is discontinued by closing stopcock, 2, the slight vacuum in the system is equalized by connecting the safety trap with atmosphere through the outlet in the bottom of the three-way stopcock, 16a. The reaction flask, 15, is disconnected from the apparatus and into it are transferred approximately 5 g (more or less, according to carbonate content) of air-dried sample weighed to the nearest 0.001 g. The sample in the reaction flask is then covered with 50 ml of carbon dioxide-free distilled water, added 1 per cent of a wetting agent, for example aerosol, shaken vigorously to insure a thorough wetting of the sample; then any adherent sample material is washed

from the walls of the flask into the mixture using little amount of CO₂-free distilled water, and the reaction flask is connected to the apparatus; the glass beads in the U-tube, 10, are drenched with concentrated sulfuric acid before each experiment.

In order to fill the apparatus with carbon dioxide free air about 1500 cu cm of purified air is aspirated through the apparatus at a rate of 50 to 75 ml per min. Then the aspiration is discontinued, the CO₂-absorption tube, 7, is removed from the train, wiped off with a chamois or chemically clean, lint-free cloth, slightly damp with water¹ and allowed to stand for 15 min in the balance case, weighed to the nearest 0.1 mg, and connected to the train again. Aspiration of purified air through the system is repeated for another 10 min at the rate of 50 to 75 ml per min, until the weight of the absorption tube is constant within 0.0003 g.

With the apparatus free of carbon dioxide and the weight of carbon dioxide absorption tube known, the analysis of the sample may be started. The aspiration system is set in operation again, and hydrochloric acid is admitted carefully from the tap funnel into the reaction flask, at such a rate that only a slow evolution of carbon dioxide is taking place. Discretion should be used in

avoiding too rapid flow exceeding the capability of the purification train to remove interfering compounds and the absorption tube to absorb entering CO₂ completely. When the evolution of CO₂ appears to have subsided, heat is applied to the reaction flask to bring the contents of it to the boiling point. To drive the reaction to completion, and to be certain that all evolved carbon dioxide is swept into the absorption tube, the contents of the reaction flask are gently boiled for approximately 30 min.

The source of heat is then removed, aspiration of air discontinued, the absorption tube disconnected from the train and weighed to the nearest 0.1 mg, taking precautions as described before. The difference in weight of the absorption tube, 7, previous to and following the liberation of carbon dioxide from the sample, represents the weight of carbon dioxide evolved.

Calculation

The percentage of carbon dioxide shall be calculated as follows:

$$\text{Carbon dioxide, per cent} = \frac{W}{C} \times 100$$

where:

W = increase in weight of CO₂ absorption tube, and

C = weight of air-dried sample.

Carbon dioxide in "moisture-free" sample =

$$\text{Carbon dioxide in "air-dried" sample} \times \frac{100}{100 - \text{moisture}}$$

Reporting of Results

The results of gravimetric determination should be reported to the second decimal place.

Volumetric Method, Employing Titration (8,9)

Several authors employing the conventional procedure for decomposition of carbonates present in solid fuels by action of diluted mineral acid have recommended the absorption of evolved CO₂ in known amount of 0.10 N barium hydroxide taken in excess; the excess is determined titrimetrically employing 0.10 N HCl and phenolphthalein as indicator.

W. Lange and W. Winzen (9) carried out the determination of carbonate CO₂ content in several coal and other samples employing (1) gravimetric, (2) manometric (or pressometric), and (3) titrimetric methods; they found the last to be superior. Time did not permit duplication and detailed investigation of the apparatus recommended by W. Lange and W. Winzen; instead, the

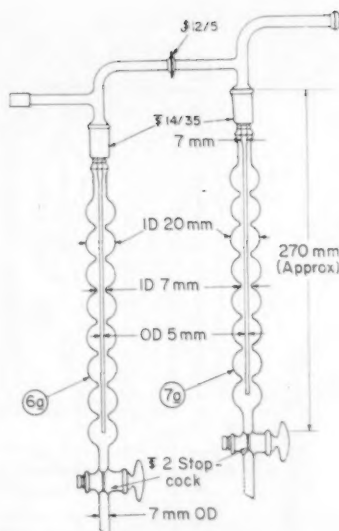


Fig. 2.—Glass vessels used for determination of CO₂ employing apparatus shown in Fig. 1, modified for titrimetric procedure.

(6a) Bulb-tube, charged with 0.10 N barium hydroxide; used as guard tube instead of item 6, Fig. 1.

(7a) Bulb-tube, charged with 0.10 N barium hydroxide; used as CO₂ absorption tube instead of U-tube, 7, Fig. 1.

final stages of the gravimetric method employed in this study were replaced by volumetric analysis in order to study titrimetric procedure.

The apparatus as employed for determination of carbonate CO₂ by the titrimetric method was assembled as shown in Fig. 1, except that the absorption tube, 7, and guard tube, 6, were replaced by two bulb tubes as shown in Fig. 2, 7a and 6a, respectively.

Procedure

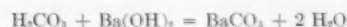
The air dried sample, 1 to 2 g (more or less, according to carbonate content) is weighed to the nearest 0.001 g, transferred into the reaction flask, covered with 50 ml of distilled CO₂-free water containing one per cent wetting agent, shaken, and the reaction flask is connected to the train. In order to fill the system with CO₂-free air, a stream of purified air is drawn through the apparatus in the same manner as described in the procedure for gravimetric determination of CO₂. Then the corresponding stopcocks are closed, the bulb-tubes 7a and 6a of Fig. 2, are disconnected from the train and in each of them is admitted 20 ml of 0.10 N Ba(OH)₂ solution employing a standard buret. Both tubes are then connected again to the train.

Air is again aspirated through the apparatus at the rate of 50 ml per min;

50 ml of 5 N hydrochloric acid is carefully admitted into the reaction flask through the tap funnel, and the reaction flask is gradually heated so that about 15 min are required to bring its contents to boiling point. A gentle boiling is maintained for another 30 min during which time about 1500 to 2000 cu cm of purified air is aspirated through the apparatus. The current of air is then discontinued and the tubes 7a and 6a of Fig. 2, are disconnected. The content of the bulb-tube, 7a, is quantitatively transferred into an Erlenmeyer flask and the excess of 0.10 N barium hydroxide solution is titrated with 0.10 N HCl with phenolphthalein as an indicator, until the content becomes colorless. The first discharge of the pink color is taken as the end point of the titration.

Whenever formation of barium carbonate was observed in the guard tube 6a (Fig. 2), the treatment of its contents was similar to that of the contents of the bulb-tube, 7a. Formation of barium carbonate in guard tube, 6a, was observed when a weight of 0.0250 g of cp CaCO₃, corresponding to 11 mg of CO₂ was taken, thus indicating that the amount of 11 mg of CO₂ liberated from the sample and carried through the apparatus under conditions of experiment is exceeding the capability of the absorption tube, 7a, to absorb entering CO₂ completely. When the amount of CaCO₃ was reduced to 0.0125 g, corresponding to 5.5 mg CO₂, no trace of barium carbonate was observed in the guard tube, 6a, which had been placed as a safeguard.

The free carbonic acid present in a diluted aqueous solution reacts according to this equation:



Since 2 moles of HCl are equivalent to 1 mole of CO₂, 1 ml of 0.10 N HCl is equivalent to 0.0022 g of CO₂.

Calculation

The carbonate CO₂ content is calculated as follows: carbonate CO₂ content in weight per cent (on air-dried basis) =

$$\frac{(a - b) \times 0.0022}{C} \times 100$$

where:

a = ml of 0.10 N barium hydroxide solution placed in absorption tube,

b = ml of 0.10 N HCl required for titration of residual Ba(OH)₂ after absorption of the CO₂ from the sample, and

C = weight of air-dried sample in g.

Gas-Volumetric Method (3, 8, 13)

The determination of carbonate CO_2 by gas-volumetric method is made as follows: the gases evolved from a weighed sample by action of mineral acid in a closed system are collected over mercury, measured at a known pressure and temperature, and forced through a solution for absorbing CO_2 usually potassium hydroxide. The difference in the volume of the gases which occurs previous to and following the absorption of CO_2 represents the volume of CO_2 released from the sample under investigation. The weight of the CO_2 is computed from its volume. The CaCO_3 or sodium carbonate are used to estimate the accuracy of the method and to determine the correction factor.

The apparatus employed for determination of carbonate- CO_2 by gas-volumetric method is shown diagrammatically in Fig. 3. It is assembled employing selected glass vessels and accessories found satisfactory in the conventional volumetric type of apparatus for analysis of gases by the volumetric-chemical method which shall comply with ASTM Method D 1136.⁴

Procedure

A weighed amount of air-dried sample, estimated to yield by decomposition upon action of hydrochloric acid at least 10 ml of CO_2 , is quantitatively transferred into the reaction tube, 3 (Fig. 3), together with approximately 0.03 grain of iron filings. The sample is covered with several drops (5 to 10) of a wetting agent⁵ and 5 to 10 ml mercuric chloride (HgCl_2) 5 per cent solution. In order to wet the sample thoroughly, the test tube is shaken; then any adherent sample material is washed from the walls of the reaction tube into the mixture using little amount of CO_2 -free distilled water. The reaction tube, 3, is connected to the apparatus as shown in Fig. 3. Then 10 ml of 37.5 per cent hydrochloric acid is placed in the separatory funnel, 2, and about 200 ml of distilled water in separatory funnel, 1.

The assembled apparatus is tested for gas tightness, and when it is found to be gas tight, when the potassium hydroxide solution in the carbon dioxide absorption pipet, 13, is at the mark near stopcock, 13a, and when mercury level in the gas measuring buret is at the mark near stopcock 8a (Fig. 3), the determination is begun. Stopcock, 8a, is turned to connect the gas measuring buret, 8, with

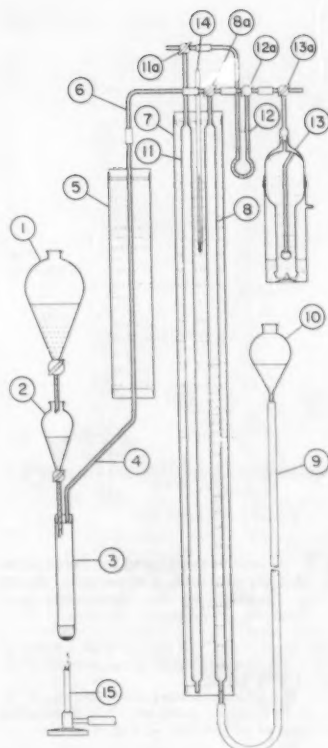


Fig. 3.—Diagram of apparatus for gas-volumetric determination of carbonate CO_2 , consisting of:

- (1) 300 ml separatory funnel charged with 200 ml distilled water.
- (2) 100 ml separatory funnel charged with 10 ml of 37.5 per cent HCl.
- (3) 22 mm \times 170 mm reaction tube containing sample, iron filings, HgCl_2 , and a wetting agent.
- (4) 8 mm OD glass tubing.
- (5) Water jacket.
- (6) 8 mm OD capillary tubing.
- (7) Water jacket.
- (8) Gas measuring buret with three-way stopcock, 8a, at top.
- (9) Rubber tubing.
- (10) Leveling bulb containing mercury.
- (11) Compensating buret with three-way stopcock, 11a, at top.
- (12) Manometer with three-way stopcock, 12a.
- (13) CO_2 -absorption pipet charged with KOH solution with three-way stopcock, 13a, at top.
- (14) Thermometer.
- (15) Gas burner.

⁴ Since the absorption of CO_2 requires a short period of time, the use of compensating buret, 11, is not necessary.

the capillary tubing, 6. The acid is then admitted drop by drop into the reaction tube, 3, at such a rate that evolved gas does not carry entrained liquids and solids upward, depositing them on the walls of the reaction tube. When the reaction appears to have stopped, the small gas burner, 15, is lighted and the reaction is driven to

completion by gently boiling the contents of the reaction tube, 3, for 2 to 5 min. The burner is then removed and all of the gases are forced over into the measuring buret by the following procedure: distilled water from the separatory funnel, 1, is run into separatory funnel, 2, and on into reaction tube, 3. The leveling bulb, 10, is lowered, creating a partial vacuum which lifts liquid up through the tubing, 4, and, 6, displaces all the gaseous products into the gas measuring buret, 8. The stopcock, 8a, is then closed to tubing, 6, and opened to the manometer, 12.

To insure that all readings of gas volume are made at the same pressure, the pressure of gas in the measuring buret, 8, before each reading is adjusted to the pressure of the compensating buret, 11, by connecting both burets with manometer, 12, and balancing the levels of colored confining solution in the manometer arms by raising or lowering the leveling bulb, 10. However, considering the short period required for absorption of CO_2 , all readings of gas volume may be made at atmospheric pressure, for this purpose connecting one arm of the manometer direct to atmosphere, not to compensating buret, and balancing the levels in the manometer arms as before.

The volume of gas, the barometric pressure, and the water temperature in the water jacket, 7, are read and recorded. After measuring the volume of the gas, it is passed at least three times through the potassium hydroxide solution contained in the carbon dioxide absorption pipet, 13, by raising and leveling the leveling bulb, 10, and the volume of gas in the measuring buret, 8, is measured as before. The absorption of the CO_2 is checked by once again passing the gas through the potassium hydroxide solution, as before, and the volume remeasured. If the volumes are equal, the gas is considered to be CO_2 -free. The difference in volume of the gas which occurs previous to and following the absorption of CO_2 represents the volume of CO_2 .

Calculation

First, the volume, V , of the CO_2 measured at, t , deg Cent and B mm pressure, moist, is reduced to 0 C and 760 mm pressure by the following formula:

$$V_0 = \frac{V(B - W) \times 273}{760(273 + t)}$$

where W represents the tension of aqueous vapor expressed in millimeters of mercury.

Since the gram-molecular volume of carbon dioxide is 22.261, 1 ml CO_2 weighs $44.01/22,260 = 0.001977$ g at 0 C

⁴ Method for Analysis of Natural Gases by the Volumetric-Chemical Method (D 1136-53), 1955 Book of ASTM Standards, Part 5, p. 1162.

⁵ In this study a 10 per cent Aerosol solution was employed as wetting agent.

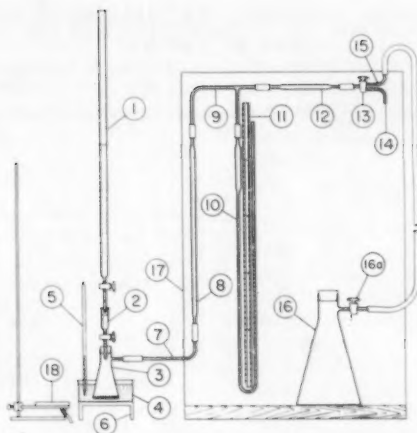


Fig. 4.—Diagram of apparatus for pressometric determination of carbonate CO₂, consisting of:

- (1) Buret charged with 5 N HCl, with 1 per cent wetting agent.
- (2) Small separatory funnel charged with exactly 2 ml of the 5 N HCl.
- (3) 50 ml reaction flask, suction type of Pyrex glass with a side arm connected to the train with flexible pressure tubing.
- (4) Water bath.
- (5) Thermometer.
- (6) Stand.
- (7) Capillary tubing connected to the train with flexible pressure tubing.
- (8) Drying tube, about 5 mm bore and about 20 cm long charged with silica gel.
- (9) Capillary connecting T-piece.
- (10) Manometer gage consisting of capillary U-tube of 1 mm bore, each arm being about 45 cm long, with the bore of one arm (connected to the T-piece) enlarged to 4 mm over a length of 7 cm.
- (11) Adjustable scale, graduated in cm and mm.
- (12) Guard tube, 10 cm long and 5 mm bore, charged with silica gel.
- (13) Three-way stopcock.
- (14) Connection to the atmosphere.
- (15) Connection to the vacuum.
- (16) Evacuated 2000 ml suction flask, used as source of vacuum.
- (17) Mounting board.
- (18) Electric hot plate.

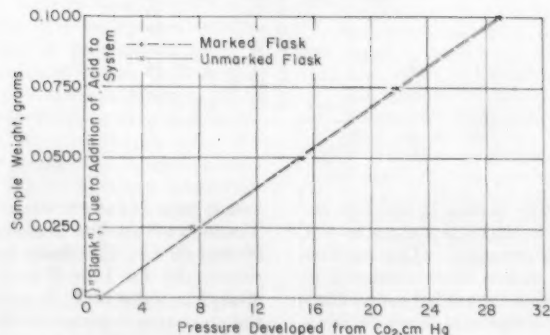


Fig. 5.—Standardization chart obtained by plotting pressure in cm Hg versus weight of cp calcium carbonate in g treated with HCl in two different reaction flasks.

and 760 mm, $V_0 \times 0.001977$ g represents the weight of carbon dioxide evolved.

Carbonate-CO₂ content in weight per

$$\text{cent (on air-dried basis)} = \frac{V_0 \times 0.001977}{a}$$

where a is the weight of the air-dried sample in grams.

For correction of the results obtained according to the above procedure, a factor is obtained by analyzing a known sample such as cp calcium carbonate or a standard sample from the National Bureau of Standards.

Pressometric Method (2, 3, 6, 8, 11)

The determination of CO₂ according to pressometric method is based upon measuring the change in pressure resulting from the decomposition of carbonates by action of hydrochloric acid in a calibrated system of constant volume. The mercury level in the manometer of the system is read and recorded. The change in height of the mercury level in the manometer is directly proportional to the amount of CO₂ generated in the reaction flask. The use of a factor-weight of material is recommended so that 10 mm net displacement of mercury in the calibrated manometer tube will correspond to 0.1 per cent of CO₂ in the sample under investigation.

The apparatus employed for pressometric determination of CO₂ is shown in Fig. 4.

Calibration

After testing for gas tightness, the apparatus must be calibrated at a specific temperature with a known sample in order to determine the net displacement of the mercury level. Several calibration tests should be made and the average from at least four determinations taken. The calibration tests must be repeated from time to time.

Note.—In this study cp calcium carbonate was used for calibration in amounts of 0.0250, 0.0500, 0.0750 and 0.1000 g. A blank test was also performed to determine effect of the mercury level in the manometer, 10 (Fig. 4), when 2 ml of HCl only (no carbonate sample) were admitted into reaction flask, 3.

The different weight values used in calibrating the apparatus versus the displacement of mercury in millimeters, observed in the manometer at the same temperature, were plotted. A curve drawn through these experimental points was a straight line. This confirms that the change of the mercury level in the manometer is directly proportional to the amount of calcium carbonate used.

Figure 5 shows curves obtained by

plotting experimental results by employing two different reaction flasks with the same apparatus, adjusting the internal capacity by adding or removing glass beads.

In calibrating the apparatus at 23 C, with 0.1000 g of powdered calcium carbonate containing 0.044 g of CO₂ the evolved CO₂ caused an average upward movement of 29.6 cm in the mercury level of the manometer, 10 (Fig. 4), this value being designated as gross displacement x . A blank test using 2 ml of acid only (no calcium carbonate), raised mercury level 1.2 cm, y . Thus, net displacement is $x - y = 29.6 \text{ cm} - 1.2 \text{ cm} = 28.4 \text{ cm}$.

Calculation of Factor-Weight to be Taken

It is recommended that a factor-weight of material be used so that 1 cm net displacement will correspond to 0.1 per cent of CO₂ in the sample. In this particular case 0.1000 g of CaCO₃ caused a net displacement of $(x-y) = 28.4 \text{ cm}$. Then factor-weight in grams is calculated:

$$\frac{0.044 \text{ g} \times 1 \text{ cm}}{0.1 (x - y) \text{ cm}} \times 100 = \frac{44}{x - y} = 1.549$$

Procedure

Finely powered sample (through 60 mesh or finer) is weighed to the nearest 0.001 g and transferred into the reaction flask, 3 of Fig. 4, with a specific number of glass beads. The stem of the separatory funnel, 2, is filled with distilled water and the funnel with stopcock is connected to the apparatus by pushing the rubber stopper each time to a calibrated mark on the neck of the reaction flask, 3, in order to maintain constant volume of the apparatus. 5N hydrochloric acid with 1 per cent wetting agent is placed in the buret, 1. Stopcock, 13, is turned in such a position that tubing, 12, and, 15, is connected. Stopcock, 16a, on the vacuum bottle, 16, is carefully manipulated so that part of the gases are withdrawn from the system, lowering the mercury in the open arm of the manometer to some predetermined position on the scale, 11. The water bath, 4, is placed in position as shown. The apparatus is permitted to stand approximately 10 min in order for the system to reach equilibrium. The degree of equilibrium is indicated by the stability of the mercury level in the manometer, 10. One millimeter movement of the mercury in 10 min is the maximum allowable. Then the scale which is divided into centimeters with 0.1 subdivisions is adjusted so that its zero is level with the top of the mercury meniscus.

Exactly 2 ml of the 5N HCl from the buret, 1 (Fig. 4), are admitted into the

TABLE I.—SUMMARY OF ANALYSES DATA OBTAINED BY EMPLOYING FOUR DIFFERENT METHODS FOR THE DETERMINATION OF CO₂ CONTENT, IN PER CENT BY WEIGHT.

Sample	Engineering Experiment Station Analyst (3, 7, 8, 16)	Gravimetric CO ₂	Titrimetric CO ₂	Gas-Volumetric CO ₂	Pressometric CO ₂
Calcium Carbonate, cp.	1	43.96 43.92 avg 43.94
Calcium Carbonate, cp.	2,3	43.84 43.78 43.84 43.50 43.84	...	42.3 42.5 42.3 42.7 42.9	...
Colorado Oil Shale.....	4	avg 43.76 20.05 20.04 20.02	...	avg 42.5 19.5 19.5 19.6 19.7 19.6	20.4 20.0 19.9 20.3 20.3 20.0
Colorado Oil Shale.....	2,3	avg 20.04 19.75 19.74	...	avg 19.6 19.5 19.8 19.6 19.5 20.0 20.2	avg 20.2 19.4 19.8 19.6 19.6 19.6
Colorado Oil Shale.....	2,3 *	avg 19.75 18.27 18.30	...	avg 19.8 18.3 18.0 18.8 18.0 18.2 18.3	avg 19.6 18.2 18.5 18.2 18.1 18.2
Ohio Oil Shale.....	2,3	avg 18.29 0.04	...	avg 18.1 0.05 0.04	avg 18.2 0.08 0.07
Ohio Oil Shale.....	2,3	avg 0.04 0.44 0.39	...	avg 0.05 0.42 0.43 0.41	avg 0.08 0.40 0.50
Ohio Oil Shale.....	2,3	avg 0.42 0.09	...	avg 0.42 0.12 0.11	avg 0.45 0.10
Ohio Oil Shale.....	2,3	avg 0.09 0.06	...	avg 0.12 0.04 0.05	avg 0.10 0.08 0.06
Ohio Oil Shale.....	2,3	avg 0.06 0.04	...	avg 0.05 0.04 0.04	avg 0.07
Ohio Oil Shale.....	2,3	avg 0.04 0.09 0.11	...	avg 0.04 0.03 0.03	0.10 0.10
Ohio Coal.....	1	avg 0.10 0.04 0.04	...	avg 0.03	avg 0.10 0.05 0.05
Illinois Coal.....	1	avg 0.04 0.13 0.13	0.14 0.16	...	avg 0.05 0.13 0.14
Illinois Coal.....	1	avg 0.13 0.36 0.34 0.34	avg 0.15 0.33 0.39 0.36	...	avg 0.14 0.39 0.38
Illinois Coal.....	1	avg 0.35 0.30 0.30	avg 0.36 0.30 0.29	...	avg 0.39 0.31 0.31
Illinois Coal.....	1	avg 0.30 2.27 2.30 avg 2.29	avg 0.30 2.44 2.22 avg 2.33	...	avg 0.31 2.30 2.31 avg 2.31

small separatory funnel, 2, and into reaction flask, 3, which is shaken to wet the sample thoroughly. (The reaction flask can be shaken while connected in the train because the rubber connections on both ends of the tube, 7, are flexible.) When the reaction subsides, the water bath, 4, and the stand, 6, are removed. To drive the reaction to completion, the flask and its contents are heated for 2

min by means of an electric hot plate, 18. The contents are thus heated to between 50 and 60 C. The flask, 3, is shaken continually for 1 or 2 min, then immersed in water bath, 4, as before and the apparatus is permitted to stand for approximately 10 min during which time equilibrium is achieved. The mercury level in the manometer is read and recorded, and the blank deducted. The

temperature is recorded, and if there is a notable deviation from the standard temperature a correction is applied. The corrected net reading in decimeters gives the percentage of CO₂ in the sample under investigation.

Summary of Results

A summary of the results of analysis for CO₂, obtained employing the four different methods, in cp calcium carbonate, oil shale, and coal is given in Table I.

Conclusions

1. The results of this study indicate that the gravimetric method has a high accuracy and good reproducibility. For example, analyses of powdered cp calcium carbonate carried out by two analysts without previous experience showed an average CO₂ content of 43.76 per cent by weight, and analyses carried out in different sets of apparatus by different analysts showed an average CO₂ content of 43.94 per cent. Both results are in good agreement with the theoretical yield which in dry sample should be 43.97 per cent. The method is simple to operate and one analyst can conveniently perform weighings and operate several, at least two, sets of gravimetric apparatus simultaneously. The gravimetric method is convenient for analyzing samples with a wide range of carbonate CO₂ content.

2. In analyzing powdered cp calcium carbonate by titrimetric method, it was observed that satisfactory results may be obtained if total CO₂ content in sample is about 6 mg or less (corresponding to 13.6 mg of CaCO₃). If total CO₂ content in sample exceeds 11 mg unabsorbed CO₂ is partially carried over into guard tube when the air velocity in system is 50 ml per min, indicating that when employing the titrimetric method, the weight of the sample should be selected according to the CO₂ content, not exceeding the above limits. This method is, therefore, suitable for investigation of samples with low carbonate content. Further study is required to reach a definite conclusion as to whether this method is preferable to other methods under consideration, as proposed by W. Lange and W. Winzen (9), since the preliminary study did not confirm this.

3. The results obtained employing the gas-volumetric method were found too low, in general. If a factor obtained by analyzing known samples is applied,

good accuracy and reproducibility may be obtained, provided that the total CO₂ amount liberated from the sample is not too small. For determining the correction factor, samples of cp CaCO₃ were used to produce volumes of CO₂ of approximately 10, 20, 30, and 40 cu cm.

The gas-volumetric method gives satisfactory results for samples containing relatively large amounts of carbon dioxide, as for example, Colorado oil shale. However, this method is not suitable for coal analysis because (1) coal usually contains small amounts of carbonates, (2) the accuracy of determination of small amounts of CO₂ is limited by the readability of the gas-measuring buret, and (3) coal tends to froth and thus considering the small size of the reaction flask, requires especially careful boiling to avoid excessive frothing.

4. The pressometric method, suitable for analyzing samples with both low and high carbonate CO₂ content, is easy to operate, and it has a good accuracy. However, a somewhat time-consuming calibration is required for each new apparatus.

Continuing Study of Analytical Methods

This report embodies the results of continued investigation of various analytical methods in order to select and develop better analytical methods of fuel research. The gravimetric method used in this study is accepted by ASTM Committee D-5 on Coal and Coke, Section C of Subcommittee XXI on Methods of Analysis, as basis for the Tentative, Revision of Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271-48)⁶. The standardization procedure is in progress.

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⁶ Tentative Revision, 1955 Book of ASTM Standards, Part 5, p. 975.

BY F. WALTER
R. R. EVANS
J. WALKER
R. J. ASKEVOLD

A LARGE portion of the petroleum wax produced in the United States today is being applied to paper for use in the packaging industry. In order to aid in the evaluation of petroleum waxes as paper coatings, the Joint Technical Committee on Petroleum Waxes, of the Technical Association of the Pulp and Paper Industry and ASTM Committee D-2 on Petroleum Products and Lubricants, has developed tests for

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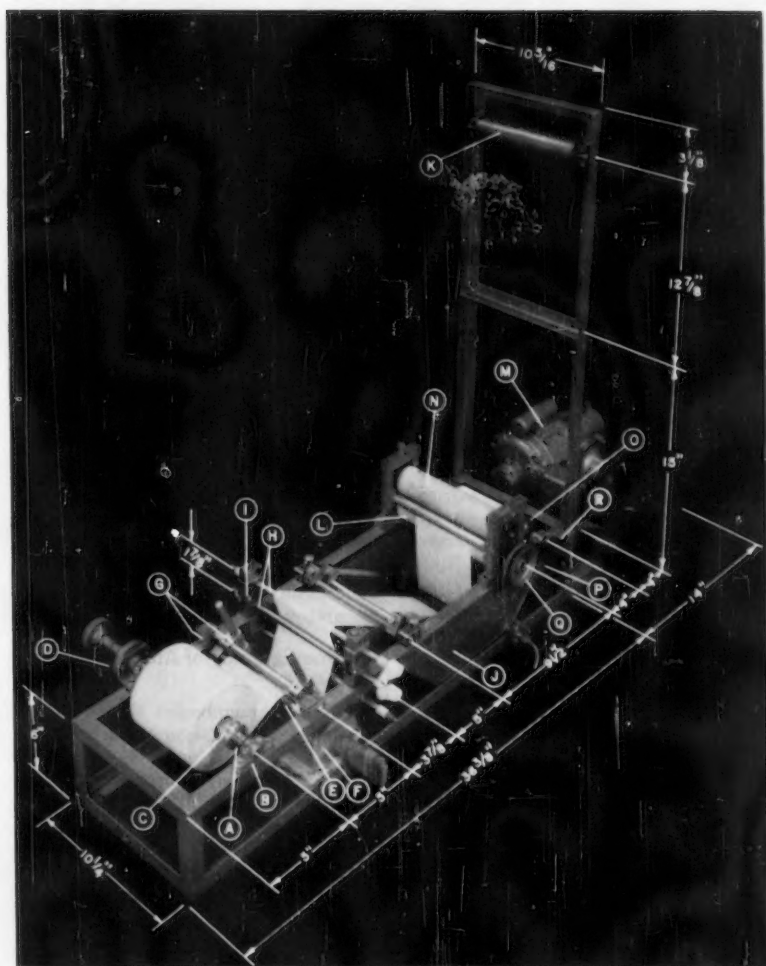


Fig. 1.—Pure Oil Laboratory Waxing Machine.



F. WALTER, now working in the Production and Transportation Division of the Research and Development Laboratories of The Pure Oil Co., Crystal Lake, Ill., was in charge of the wax testing group in the primary development of the waxing machine.



R. R. EVANS has been in the Research and Development Laboratories of The Pure Oil Co. since 1951 and is at present in charge of the wax testing group.



J. WALKER, is at present supervisor of the Physical Chemistry Section of which the wax testing group is a part. He is a member of several sections of the TAPPI-ASTM Technical Committee on Petroleum Waxes.



R. J. ASKEVOLD, director of the Analytical Research and Service Division of The Pure Oil Co.'s Research and Development Laboratories, is a member of the TAPPI-ASTM Technical Committee on Petroleum Waxes.

blocking point, sealing and laminating strength, and gloss evaluation. To carry out these tests it is essential to have available some type of apparatus permitting the application of the wax to paper in a manner similar to that used in large scale coating machines. In actual commercial operations, such coating conditions as the paper speed, the weight of wax applied to one or both sides of the paper, and the type of cooling employed may vary widely depending upon the quality of the finish required in the end product.

The Pure Oil Co. Research and Development Laboratories have developed a laboratory waxing machine to apply petroleum waxes to paper in order to carry out such performance tests as those mentioned above. The apparatus has evolved through a continuous process of change and improvement during the past four years. A dimensioned photograph of the apparatus is shown in Fig. 1. The speed of the paper can be adjusted within the range of 0 to 200 ft per min, and the wax coating weight can be varied from 0.5 to 15 lb of wax per ream of paper. Either air or water cooling of the coated sheet may be employed, and the cooling rate can be varied at will.

Description of Waxing Machine

The waxing machine was constructed using an angle-iron frame for rigidity. Spindle *A* holds the roll of paper to be coated, which can be up to 5 in. in width. The spindle is mounted on pillow block bearings, *B*, and is provided with cone retainers, *C*, to center and hold the roll of paper. An adjustable brake mechanism, *D*, controls the tension on the paper web. The brake is composed of two flat disks with adjustable spring loading which impose a drag on the unwinding paper. This aids in controlling the amount of wax placed on the paper by regulating the pressure of the paper against the doctor rods.

After the paper leaves the spindle, it passes over guide bar, *E*. This guide bar is provided with a movable sleeve consisting of a piece of brass tubing slipped over the Fisher Flexa-frame aluminum rod $\frac{1}{2}$ in. in diameter. From this guide bar the paper passes down under a second guide bar in the wax reservoir, *F*. The guide bars are held in place by Fisher Flexa-frame connectors, *G*. The wax reservoir which is made of cast aluminum and has a capacity of 200 g rests on a 250-w electric strip heater that is controlled to the desired temperature by means of a variable autotransformer. The wax temperature is measured by a thermometer inserted in the molten wax.

To remove the wax reservoir for changing samples, the set screws are loosened in the Fisher Flexa-frame connectors mounted on the angle-iron frame. This permits the upper horizontal rod which holds the guide bar assembly in place to rotate. By rotating the guide bar assembly upwards, the lower guide bar is freed from the wax reservoir. The reservoir can then be pulled out, cleaned and filled with a new wax sample.

From the wax reservoir the paper passes over two stationary doctor rods, *H*, to remove excess wax from the coated paper. The doctor rods are held in place by two Fisher Flexa-frame multi-clutch connectors, *I*, which have had the holes slotted to the outside edge and are provided with a spring clip. This permits easy installation and removal of the doctor rods. The doctor rods consist of $\frac{5}{8}$ -in. outside diameter heavy walled stainless steel tubing machined with special threads of semi-circular section to simulate winding with wire in the range of 30 to 40 B & S gage. The amount of wax left on the paper depends on the wire gage chosen. Since the thread size is inversely proportional to the gage numbers, less wax is left on the paper by the higher gage number. Pairs of rods are provided with threads corresponding to even numbered B & S wire gage sizes of 30, 32, 34, etc., to permit application of the same amount of wax to both sides of the paper. Pairs of rods in which only one of the rods is threaded are also provided for use when only one side of the paper is to be coated. The unthreaded rod removes practically all of the surface wax. Nichrome wire heaters are incorporated within the rods to provide a means for heating them to the desired temperature, and these heaters are controlled by means of a variable autotransformer. The temperature of the rods is measured by means of a thermocouple silver soldered to the outside of the rod.

From the doctor rods, the waxed paper passes through the water bath, *J*, if water cooling is to be used, or over a roller, *K*, which extends approximately 3 ft above the doctor rods, if air cooling is desired. The temperature of the water bath is adjusted by adding cold water before the run. It has been found that the temperature of the bath does not rise significantly during the short 1 to 2 min normal running time. It was originally planned to circulate coolant through the bath but this was found to be unnecessary.

The force necessary to pull the paper through the apparatus is provided by a power roll, *L*, which is rotated by a V-belt driven by a $\frac{1}{4}$ -hp geared motor, *M*,

(G.E. model- 5KC45AB781, gear model- 7G712C1, 1725 rpm, gear speed 99 to 1, gear ratio 17.5 to 1). An idler roller, *N*, on top of the power roll is spring-loaded to insure gripping of the coated paper. These drive rolls were constructed from two washing machine wringer rollers. Since the motor is a constant speed motor, the paper speed is varied by changing the pulley combinations on the drive, although a variable speed motor would accomplish the same thing. A wind-up roll, *O*, is coupled to the power roll by means of a Fusite belt, *P*, and pulleys. A metal pulley, *Q*, is attached to the power roll while a smaller rubber pulley, *R*, is attached to the wind-up roll. This allows the Fusite belt to slip on the metal pulley and automatically maintain the wind-up roll at the proper speed to take up the coated paper as it passes from between the drive rolls.

A paper slitter designed to slit the paper automatically into strips of desired width for specific tests such as blocking point can also be attached to the machine.

Operating Procedure

In setting up the waxing machine for operation, a roll of paper is positioned on the spindle, *A*, and centered in place with the cone retainers, *C*. The proper doctor rods are selected and installed. The paper is threaded through the machine as indicated in Fig. 1, except that it is not passed through the water bath, *J*, or drive rolls. The wax reservoir and doctor rod heaters are then turned on and the temperature of each is adjusted to approximately 35 F above the melting point of the wax to be tested. The wax reservoir, *F*, is then filled with the molten wax. When the wax has reached the desired temperature, as measured by a thermometer, the paper is pulled through by hand until the waxed portion has cleared the water bath. The waxed paper web is then threaded through the water bath, or over the air cooling roll, and then through the drive rolls. The web is then attached to the wind-up roller.

The machine is run until sufficient paper has been coated to use in measuring the wax coating weight. It may be necessary to make fine adjustments of this weight by tightening or loosening the brake slightly to impose more or less tension on the paper as it passes over the doctor rods, although the major factor in the adjustment of the wax coating weight is the selection of the proper doctor rod (higher gage number for less wax). When the coating weight has been adjusted, a sufficient quantity of paper for test purposes can then be waxed.

The Effect of Loading Rate on Adhesive Strength

BY FRANK MOSER AND SANDRA S. KNOELL

Results show significant differences between loading rates, testing machines, and adhesives

ASTM METHODS for testing adhesives specify that definite rates of loading should be used for evaluating adhesive bonds.¹ For example, for a crosshead movement machine, a jaw separation rate of 0.05 in. per min. is specified while for a hydraulic machine the load is to be applied at a rate of 600 lb per min. The results of strength tests obtained from the two testing machines are often confusing and are difficult to correlate. Apparently, these speeds were set arbitrarily and therefore a further study of loading rates is advisable in order to obtain comparisons between loading rates and bond strength obtained on respective machines.

Some preliminary tests were made on glass surfaces, but too much glass failure occurred giving little information on the adhesive strength. Therefore, glass was discarded and aluminum and steel were used as the bonding surfaces.

Experimental

The rate-of-loading included three adhesives, two testing machines, and two rates of loading. The arrangement of the experiment is shown in Table I and consists of 12 unit cells for comparisons between machines, rates of loading, and adhesives for each of two metals. Other variables such as the

TABLE I.—ARRANGEMENT OF UNIT CELLS FOR EACH METAL.

Metals	Testing Machines	Adhesives	Rates of Loading
M ₁ ...	T ₁	A ₁	S ₁
M ₁ ...	T ₁	A ₂	S ₂
M ₁ ...	T ₁	A ₃	S ₂
M ₁ ...	T ₁	A ₃	S ₂
M ₁ ...	T ₁	A ₃	S ₂
M ₁ ...	T ₂	A ₁	S ₂
M ₁ ...	T ₂	A ₂	S ₂
M ₁ ...	T ₂	A ₃	S ₂
M ₁ ...	T ₂	A ₃	S ₂
M ₁ ...	T ₂	A ₃	S ₂

preparation of the metal surfaces, application of bonding pressure, curing procedure, and order of testing were randomized, while the bonds were pre-

pared in blocks of 12 for obtaining the ten replications for each adhesive.

The adhesives were bonded in blocks in order to compensate for any effects that might be encountered from day to day, from atmospheric conditions, and from any personal elements which may influence the preparation and testing of the bonds.

Dillon and Baldwin Southwark testing machines (T₁ and T₂ in Table I) were used for obtaining bond strength, using the same specimen-holding jigs. No attempt was made to standardize the two type machines, with respect to equivalent loading rates in pounds per minute, but each was run at a recommended ASTM designation for the type of machine. The loading for the

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¹ Tentative Method of Testing Cross-Lap Specimens for Tensile Properties of Adhesives (D 1344-54 T). 1955 Book of ASTM Standards, Part 7, p. 1219.

crosshead machine was at 0.05 and 0.10 in. per min, while the hydraulic machine was set for 600 and 1200 lb per min (S_1 and S_2 in Table I). Adhesives of

² NBR—Nitrile Butadiene Rubber; see Recommended Practice for Nomenclature of Synthetic Elastomers and Latexes (D 1418-56 T) 1956 Supplement to Book of ASTM Standards, Part 6, p. 201.

³ The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE II.—TENSILE STRENGTH OF METAL-METAL ASSEMBLIES BONDED WITH A VINYL ADHESIVE, A_1 .

	Machine I, Dillon		Machine II, Baldwin	
	M_1 Iron	M_2 Aluminum	M_1 Iron	M_2 Aluminum
Speed 1....	3000 3600 2500 3200 2200 3200 3500 1800 3100 2700	2300 2600 1300 1600 3700 1600 3900 3800 3700 3200	2000 2100 1500 1700 1600 2200 2700 2000 2200 2400	2800 3100 2200 1200 2300 2400 2500 1900 2800 2800
Average....	2780	2770	2040	2400
Speed 2....	2900 2700 4700 3700 2700 3400 3200 3400 2600 3600	3400 2900 3000 2700 4000 4100 3800 3200 4200 3100	1600 1800 2400 2900 2200 2400 2600 2400 2200 2300	2800 1900 1500 2500 2100 3400 2700 2700 3200 3700
Average....	3290	3440	2280	2650

TABLE III.—TENSILE STRENGTH OF METAL-METAL ASSEMBLIES BONDED WITH A MODIFIED PHENOLIC ADHESIVE, A_2 .

	Machine I, Dillon		Machine II, Baldwin	
	M_1 Iron	M_2 Aluminum	M_1 Iron	M_2 Aluminum
Speed 1....	1600 3800 3100 3600 3100 3500 3200 3600 2300 3600	3600 3900 3800 3600 3800 3000 3700 3000 5100 4300	3100 2900 3300 2900 3700 3600 1900 2800 2000 1900	3200 3200 4100 2500 2400 4600 2400 3200 2500 3500
Average....	3140	3780	2810	3160
Speed 2....	5200 5200 7500 4400 4100 6300 4300 4700 4200	3300 4600 3400 3600 3800 3900 4400 4400 3800	1800 2100 1900 2600 4500 2700 3200 3500 3200	3000 2900 2600 2600 3600 4600 4400 3900 3800
Average....	4990	3900	3000	3410

both brittle and elastic types including a vinyl copolymer a poly(vinyl butyral)-phenolic, and a vinyl-NBR² types A_1 , A_2 , and A_3 respectively in Table I, were chosen for this study. Curing procedure was identical for each and consisted of air drying the film for 1 hr, followed by a 15 min unassembled heat activation at 250 F, assembled under 200 psi pressure and cured in a circulating air oven for 45 min at 300 F.

Preparation of the metal surfaces before application of adhesive was as follows: The aluminum was cleaned in a sulfuric acid-sodium-dichromate bath at 140 F according to the method proposed by the Forest Products Laboratory (1).³ The steel was prepared by smoothing over a series of metallographic emery papers ending at No. 000

TABLE IV.—TENSILE STRENGTH OF METAL-METAL ASSEMBLIES BONDED WITH A VINYL-NBR TYPE ADHESIVE, A_3 .

	Machine I, Dillon		Machine II, Baldwin	
	M_1 Iron	M_2 Aluminum	M_1 Iron	M_2 Aluminum
Speed 1....	1800 1800 1900 1800 2400 1900 2400 2400 2600 2200	1600 1600 1400 2800 1400 1600 2100 1400 1800 1900	1900 1900 1900 1100 1300 1600 1400 1700 1800 1800	1500 1400 1800 2000 1700 2100 1700 1400 1100 2000
Average....	2120	1760	1640	1670
Speed 2....	1700 1900 1700 2700 2300 1900 2300 2600 2100 2600	1600 2000 1800 2200 2100 1700 1700 2100 2000 2100	1600 1300 1600 1800 1400 1600 1700 1600 2300 1900	1600 1900 1600 1400 1700 1600 1700 1600 2400 2300
Average....	2180	1930	1700	1780

TABLE V.—COMBINED ANALYSIS OF VARIANCE.

	Sum of Squares	Degrees of Freedom	Mean Square	F_{05} Ratios
Adhesives (A).....	11 241.52	2	5620.76	158.77*
Speeds (S).....	836.26	1	836.26	23.623*
Machines (T).....	2 368.81	1	2368.81	66.915*
Metals (M).....	19.26	1	19.26	0.544
A \times S.....	258.37	2	129.18	3.649
A \times T.....	340.32	2	170.16	4.807
A \times M.....	118.62	2	59.31	1.675
S \times T.....	216.61	1	216.61	6.119*
S \times M.....	74.83	1	74.83	2.114
T \times M.....	264.61	1	264.61	7.475
A \times S \times T.....	135.96	2	67.98	1.920
A \times S \times M.....	280.69	2	140.35	3.965
A \times T \times M.....	26.71	2	13.30	0.376
S \times T \times M.....	104.09	1	104.09	2.940
A \times S \times T \times M.....	299.59	2	149.79	4.231
Residual.....	7 646.40	216	35.40	
TOTAL.....	24 232.65	239		

* Very significant.

fineness and finally wiping with trichloroethylene just before application of the adhesive. Tests were carried out according to ASTM method D 1344 for determining tensile strength.⁴

Discussion of Results

Analysis of variance techniques (2) were used to determine the effects of rate of loading on bond strength. Data obtained for tensile strength of three adhesives are given in Tables II, III, and IV, while in Table V the " F " ratios for variables at the 5 per cent significance level are given. This is referred to as the F_{05} ratio.

As the adhesive specimens were made up in blocks of 12 units, the first concern is to determine if any day-to-day effects in the preparation and testing of the adhesive assemblies show any significant differences. The " F_{05} " value calculated from the data for blocks totals gave an F_{05} (9216) value of only 1.26, whereas the " F_{05} " value for significance given in the F distribution table for 9 and 216 degrees of freedom is 2.41. Therefore, no significant difference occurred between the blocks, and for further calculation of significance the block totals are pooled with the residual for determining the effects of other variables.

High significant differences were obtained for adhesives, speeds, and testers. F_{05} values obtained were 158.8 for adhesives, 23.6 for testing speeds, and 66.9 for testing machines. Such data indicate highly significant differences, as the F_{05} values for significance for the respective degrees of freedom for each variable are 3.0, 3.84, and 3.84. The data for differences between metals show no difference in bonding strength between the metals as only a 0.55 " F_{05} " value was obtained.

Also, the interactions between variables are presented in Table V. Interactions exist between speeds and ad-

hesives, machines and adhesives, speeds and machines, and machines and metals. Second and third order interactions are present between adhesive-speed-metals and between adhesive-machines-speeds-metals. Such associations are expected due to high significance between speeds and machines. However, it was unexpected that an F_{05} ratio of 7.47 for the $T \times M$ interaction would be highly significant because no significant differences occurred between the metals. However, a further analysis of this interaction becomes imperative and is presented graphically in Fig. 1.

Comparisons of the strength obtained by the two machines with the respective metals are shown. With the vinyl copolymer adhesive, A_1 , the bond strength is much more variable on steel than on aluminum. This is more pronounced with adhesive A_1 than with the other adhesives, A_2 and A_3 . For example, bond strength of adhesive A_1 varies from 3035 psi on steel to 3105 psi on aluminum with machine T_1 , while with machine T_2 the strength for steel is 2160 psi and 2525 psi for aluminum. With adhesive A_2 , bond strengths have more variability, giving 4020 psi on steel and 3840 psi on aluminum for machine T_1 . The values on machine T_2 were 2905 psi on steel and 3205 psi on aluminum. However, with the vinyl-NBR adhesive, A_3 , a wider range in strength occurred between the two machines for steel than with the aluminum, giving 2150 psi for steel and 1845 psi on aluminum with Dillon, while the Baldwin gave 1670 psi for steel and 1740 psi for aluminum.

These differences in behavior between the aluminum and steel as bonding surfaces may help explain the $M \times T$ interaction. The Dillon machine gave higher values than the Baldwin. However, the Baldwin was the more consistent machine, always giving higher values for aluminum-aluminum bonds than for steel-steel bonds, while with the Dillon, aside from the vinyl adhesive, the tendency was the opposite, giving higher strengths on steel than on aluminum with the modified phenolic and the vinyl-NBR adhesives. Such behavior caused the difference between metals to be minimized, and these averages appeared to balance each other so that no significance was found between metals by the "F" ratios.

Loading rate and bond strength as obtained with the three adhesives are presented in Fig. 2 on each metal. The high-modulus type adhesives A_1 , A_2 show considerable variation between the standard and double rate of loading, while a low-modulus adhesive A_3 shows very little difference in strength due to

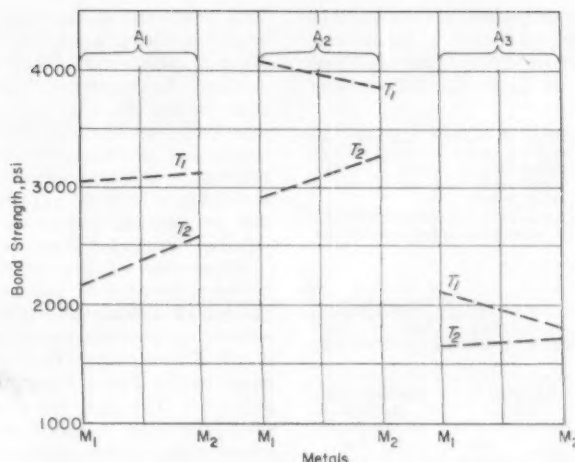


Fig. 1.—Metal and testing machine effect on bond strength.

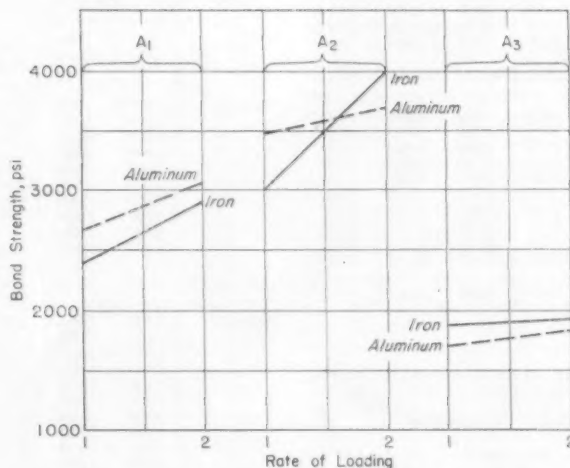


Fig. 2.—Bond strength versus rate of loading for two metals.

rate of loading. With both metals the strength of adhesive increased with the double rate of loading. The various adhesives show preferential adhesion for the respective metal surface. For example, the vinyl copolymer with aluminum gave a bond strength of 2410 psi for the standard loading rate and increased to 3055 psi with the double rate. Approximately the same adhesion was secured with steel surfaces with this adhesive. However, with the modified phenolic type adhesive, the steel surface gave the greater increase for the double rate of loading from 2975 psi to 3950 psi, while aluminum gave only a slight in-

crease ranging from 3470 psi to 3655 psi for standard and double rates of loading. These differences in adhesion may be associated with the physical properties of the two metals. With highly elastic adhesives, the differences in adhesion are reduced, giving a smaller variation between the metal surfaces, and also between rates of loading.

Further comparisons of bond strengths are given in Fig. 3 for the rate of loading on respective testing machines. The Dillon machine T_1 gave the higher strength and appears to have more rapid rise in tensile strength when the rate of loading is double. In the case

of the vinyl copolymer adhesive, A_1 , a value of 2775 psi was obtained for the standard rate of loading, while the double rate was 3365 psi. The Baldwin machine, T_2 , gave a uniform increase from 2200 psi to 2465 psi when the loading was increased from 600 to 1200 lb per min. For very high modulus adhesives, the rise is greater on the Dillon machine, T_1 , for the double rate of loading. In this case the standard rate gave 3460 psi, while the double rate yielded 4400 psi. However, on the Baldwin T_2 , a gradual increase from 2985 psi to 3205 was obtained. With the low-modulus adhesive A_3 , the vinyl-NBR type, the slope of the curve was almost identical for both machines, but the Dillon, T_1 , was approximately 300 psi higher in strength at both rates of loading.

The higher tensile strength of the three adhesives on the Dillon machine, T_1 , may be attributed to the fact that no attempt was made at the outset of the experiment to standardize the two rates of loading in psi per min. Apparently the 0.5-in. rate of separation applies the load at a more rapid rate than 600 psi per min. Several checks were made on the Dillon machine T_1 using a stop watch to obtain loading rate in psi per min. The results clearly indicate that load on a psi basis, the 0.05 and 0.10 in. per min. for the jaw separation on the Dillon T_1 , exceeded the 600 and 1200 psi used for testing on the Baldwin, T_2 . These results of the two machines confirm our assumption that higher values would be expected with the Dillon, T_1 . Perhaps a further study would be advisable in which the two loading rates were standardized on a psi per min basis.

Curves are presented for the two rates of loading in Fig. 4 in order to correlate the results of one machine with the other. The curve would be more complete if additional adhesives were included so as to have mean strengths at 1000 psi and above 5000 psi. However, the three adhesives have established a trend when the value of one machine is plotted against the other. At the low rate of loading, the curve indicates that a quadratic effect is occurring while at the double rate of loading a linear relation is being approached.

Summary

An analysis of variance was used to determine the effect of loading rates

upon the strength of adhesives. The results show very significant differences between loading rates, testing machines, and adhesives. With high-elastic-modulus adhesives, $A_1 + A_2$, more difference in strength is obtained from the increased rate of loading, while with a low-modulus adhesive A_3 only a small increase in strength is obtained for the higher loading rates.

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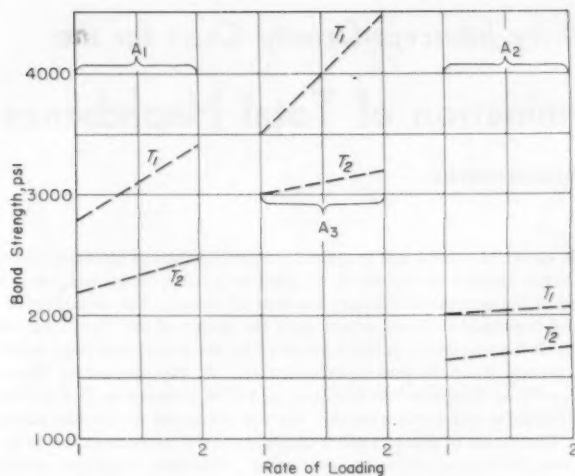


Fig. 3—Bond strength versus rate of loading for two testing machines.

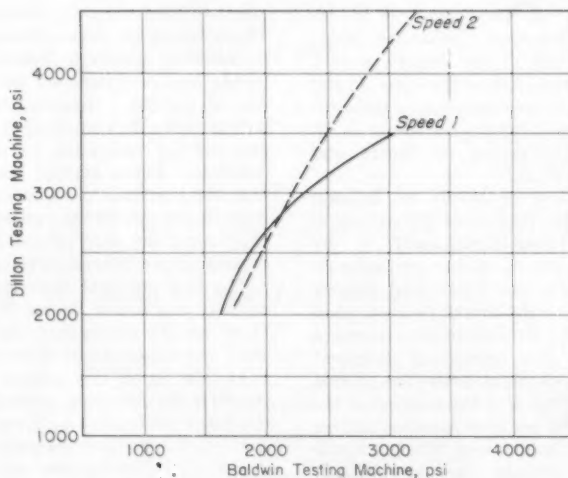


Fig. 4.—Testing machine comparison.

Refractivity Intercept-Density Chart for the Determination of Total Naphthenes in Gasoline

By SIGURD GROENNINGS

The refractivity intercept graphical method for determination of the total naphthene content of distillates of gasoline boiling range has been employed in the petroleum industry for over 20 years. The reliability of the method depends to a large extent upon the design of the chart from which the result is computed. A chart prepared by the author has been available on a private basis to several laboratories. At the request of Research Division IV on Hydrocarbon Analysis, of ASTM Committee D-2 on Petroleum Products and Lubricants this chart is presented for broader information. Evaluation of this and other charts, in use or recommended, is based on a very limited number of experiments. However, from such evidence as is available, very good reliability and scope of applicability may be claimed for this chart.

APPPLICATION of the refractivity intercept (refractive index minus one half of the density, $n-d/2$) to the determination of the ratio of total naphthenes to total paraffins in the saturates portion of lighter petroleum distillates was introduced by Kurtz and Ward in 1936 (1).¹

The method is briefly as follows: The saturates portion of the sample is isolated (chromatographically or by acid absorption of olefins and aromatics), its refractive index and density determined to the fourth decimal place at 20 C, and the refractivity intercept calculated. The refractivity intercept and density of the saturates are plotted on a graph depicting the position of the curves for 100 per cent paraffins and for 100 per cent naphthenes, which are connected by straight lines representing saturates of approximately equal boiling points. The naphthene, or inversely, the paraffin content of the saturates portion is then calculated from the position of the point by linear interpolation on the chart, and, in turn, calculated on the basis of the original gasoline from the known saturates content of the latter.

It is evident that the reliability of such a chart depends essentially upon the positions of the 100 per cent par-

affin and 100 per cent naphthene curves. These curves are drawn through points representing available literature data, taking into consideration the distribution of isomers. However, this distribution varies depending upon the original and the processing history of the distillate. For a general chart, therefore, the positions of all-purpose curves must be estimated from available knowledge regarding the preponderance of certain types of compounds over others.

The first practical description of the method was issued during World War II as ASTM Emergency Method ES-45a,² and subsequently several publications bearing on this subject have appeared in the literature, as enumerated in the latest publication by Wood *et al.* (2) In the latter, it is pointed out that better results should be expected when analyzing distillation fractions of the gasoline and employing charts applicable to each particular fraction. Attention is also called to the fact that in the higher boiling region of gasoline, above about 150 C, bicyclic naphthenes appear, and since their refractivity intercept and density differ appreciably from those of the monocyclic naphthenes, the usability of a chart for this region will be doubtful. Nevertheless the original general chart of Method ES-45a has been used extensively for analyses not only of lower boiling fractions but of the whole gasoline as well, because the method is very simple as it requires no special equipment, and the error due to the presence of bicyclic naphthenes will be less pronounced when based on the whole gasoline.

Shortly after the publication of the Emergency Method, the present author constructed a chart with the paraffin

and naphthene curves in somewhat different positions. It is felt in many laboratories that this chart gives better results than the original, although there is as yet no entirely satisfactory evidence by which to prove it. Meanwhile this chart is being used extensively, and for this reason Research Division IV of ASTM Committee D-2 on Petroleum Products and Lubricants has requested that it be published in the ASTM BULLETIN for future reference.

Data for Chart

Figure 1 shows a plot of refractivity intercept *versus* density of the paraffins and monocyclic naphthenes from available literature data, including the most recent, to improve on the trend. Some of these data are less reliable than others, but not sufficiently so to affect the general picture appreciably. The sources of this information are listed in Table I. It is interesting to note that one may readily discern in this graph a trend toward convergence of all points at the limiting values for refractivity intercept and density, 1.0495 and 0.8510, respectively; this is particularly the case with normal and near normal (2- and 3-methyl) paraffins and with normal alkyl cycloparaffins which progress in linear succession.

The dashed lines indicate the more or less arbitrarily chosen loci for 100 per



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¹ The boldface numbers in parentheses refer to the list of references appended to this paper.

² Emergency Method of Test for Olefins, Aromatics, Paraffins, and Naphthenes in Aviation Gasoline (without distillation into fractions) (ES-45a), 1945 Book of ASTM Standards, Part 3, p. 153.

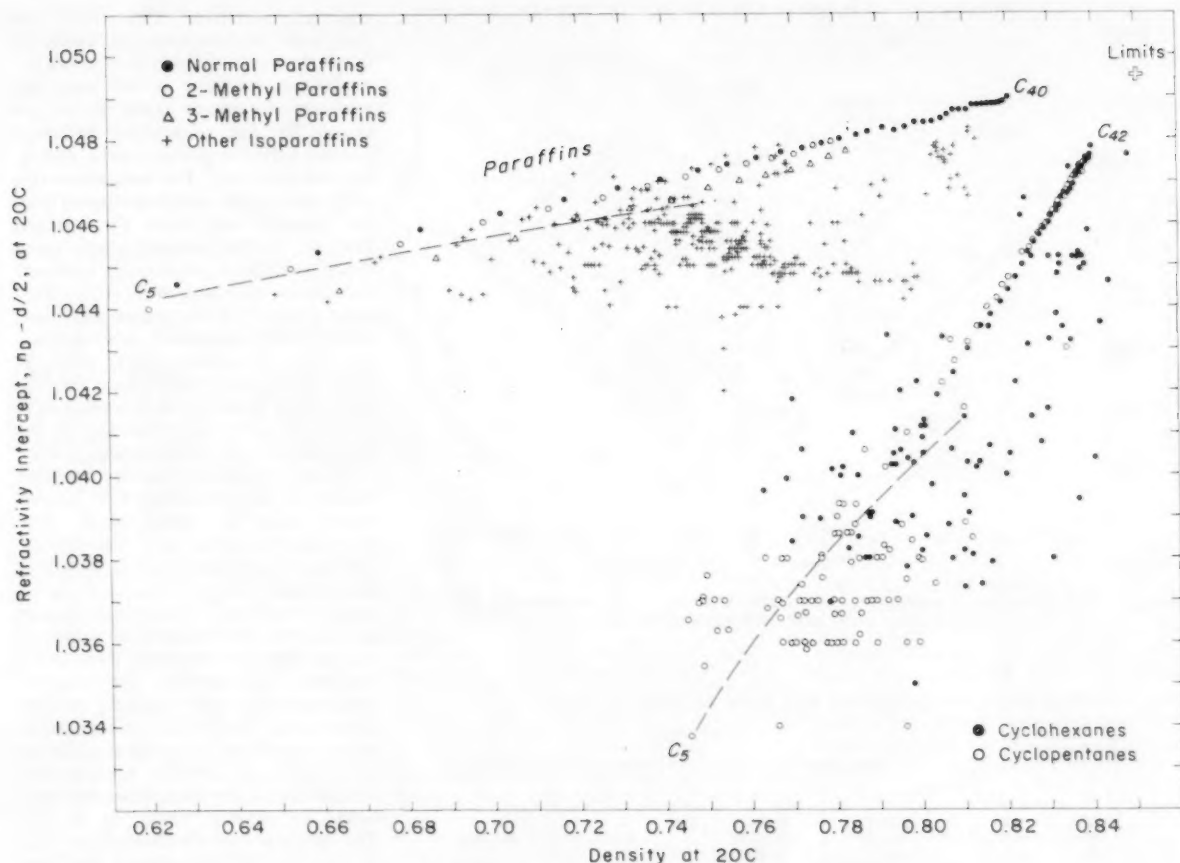


Fig. 1.—Refractivity intercept *versus* density of saturated hydrocarbons.

cent paraffins and 100 per cent naphthenes in the gasoline boiling range, 30 to 200 C. The position of the paraffin line is governed by the rather well founded premise that the normal and singly branched paraffins predominate, the content of more than singly branched paraffins tending to increase somewhat at the upper end. The naphthene line, starting at the cyclopentane point, first follows the normal cycloparaffins, later to deviate from the latter in the direction of the center of the widely spread points in the upper region; this curvature is furthermore designed to compensate in part for the presence of bicyclic naphthenes which have relatively low refractivity intercepts and high densities.

The Chart

The chart illustrated in Fig. 2, incorporates the 100 per cent paraffin and naphthene curves from Fig. 1, with divisions into approximate boiling point sections, and connected by straight lines. The purpose of these lines is merely to serve as directional guides for

TABLE I.—SOURCES OF DATA FOR FIG. 1.

Carbon Atoms	Paraffins	Number of Compounds
C ₅ to C ₁₈	Normal and 2- and 3-methyl paraffins. Smoothed values based on critical study of available physical constants, 1940 (3)	45
C ₅ to C ₁₁	Isoparaffins and	306 391
C ₁₀ to C ₁₆	Normal paraffins, API, 1952-1956 (4)	
C ₁₂ to C ₂₂	Isoparaffins, M. P. Doss, 1943 (5)	40
Naphthenes		
C ₅ to C ₄₁	Normal alkyl cyclopentanes and	116
C ₆ to C ₉	Alkyl cyclopentanes, API, 1954-1956 (4)	
C ₁₀ to C ₂₄	Alkyl cyclopentanes, M. P. Doss, 1943 (5)	7
C ₆ to C ₁₂	Normal alkyl cyclohexanes and	64
C ₇ to C ₉	Alkyl cyclohexanes, API, 1954-1956 (4)	
C ₁₀ to C ₃₁	Alkyl cyclopentanes and alkyl cyclohexanes, M. P. Doss, 1943 (5)	54
		Total 632

measurement of the distance from the point representing the sample to the paraffin line and the distance between the basic curves for calculation of the naphthene content. This calculation is facilitated by dividing the guiding lines into ten equal parts, as paraffins and naphthenes mix very nearly linearly in respect to refractive index and density.

The coordinates for construction of a working chart are given in Table II; it should be drawn on 300 by 400 mm graph paper so that refractivity intercepts and densities can be plotted and read to the fourth decimal place. (The table gives refractivity intercepts to the fifth decimal place to facilitate construction of the curves.)

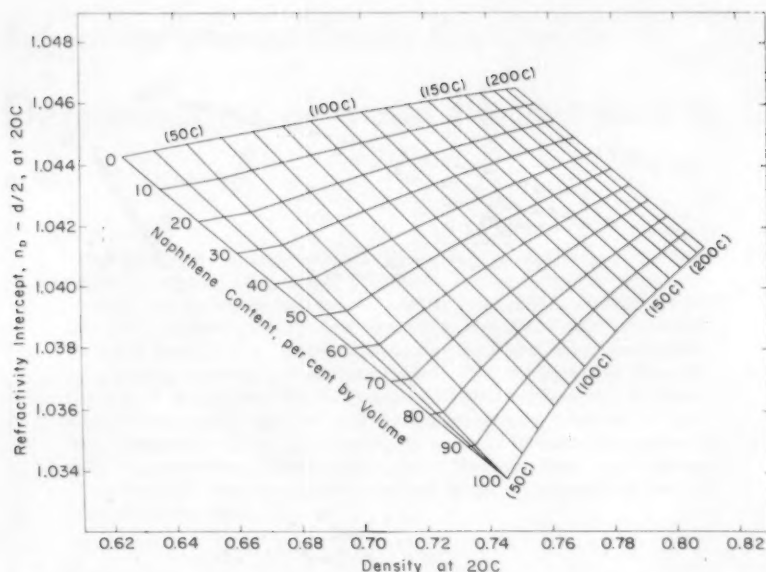


Fig. 2.—Determination of naphthene and paraffin contents of the saturated portion of gasoline from refractivity intercept and density.

TABLE II.—COORDINATES FOR CONSTRUCTION OF FIG. 2.

Approximate boiling point, deg Cent	Connecting ^a			
	Points on Paraffin Curve		Points on Naphthene Curve	
	$n - d/2$	d	$n - d/2$	d
30.....	1.04426	0.6235	1.03376	0.7454
40.....	1.04448	0.6351		
50.....	1.04470	0.6462	1.03462	0.7505
60.....	1.04490	0.6567		
70.....	1.04509	0.6667	1.03542	0.7558
80.....	1.04527	0.6761	1.03617	0.7610
90.....	1.04543	0.6850	1.03685	0.7663
100.....	1.04559	0.6935	1.03748	0.7715
110.....	1.04574	0.7015	1.03806	0.7766
120.....	1.04587	0.7089	1.03859	0.7816
130.....	1.04599	0.7158	1.03907	0.7862
140.....	1.04610	0.7222	1.03952	0.7905
150.....	1.04620	0.7282	1.03992	0.7946
160.....	1.04628	0.7335	1.04028	0.7983
170.....	1.04635	0.7383	1.04061	0.8018
180.....	1.04641	0.7426	1.04089	0.8046
190.....	1.04646	0.7462	1.04113	0.8071
200.....	1.04650	0.7495	1.04133	0.8092

^a Lines connecting the paraffin and naphthene curves are divided into ten equal parts.

Accuracy

Unfortunately no reliable information regarding the dependability of the present or the original Method ES-45a chart as applied to full range gasolines is available. The reason for this is that there are no entirely acceptable criteria on accuracy. Mass spectrometry appears to be the most promising, however. This would entail a considerable amount of work because for best results the analyses would most likely have to be carried out on numerous distillation

fractions and from many types of gasoline. However, such testing should be undertaken if one wishes to ascertain whether this simple routine method does yield reasonably useful results, at least when applied to whole gasolines of low bicyclic naphthene contents.

For the lower boiling range, below 150 C, on the other hand, some information is available. Thus Wood, *et al.*, in a recent publication (2) give the results of reliability tests of their two recommended special boiling range charts, one for wide range fractions, 60 to 135 C, and one for narrow fractions (essentially C_6), 60 to 86 C, using naphthene contents determined by infrared spectrom-

etry as reference. The results of their tests are reproduced in Table III which shows the differences between the naphthene contents of the saturates portions of various distillates as obtained by the refractivity intercept method with the special charts, and by infrared analyses. For comparison this table includes the results computed from the present and from the original Method ES-45a general chart using Wood's values for refractivity intercept and density. It may be concluded from these results that the special charts are, on the whole, somewhat more dependable than the present chart, which, in turn, is somewhat better than the ES-45a chart. However, this conclusion is contingent upon the reliability of the infrared data used as standards.

Another reliability test is presented because of the probability that the reference value is rather good. The American Petroleum Inst. recently undertook a cooperative test of a California reformer charge stock, applying all available methods. These include mass and infrared spectrometry of numerous sharply distilled fractions, refractivity intercept, and catalytic dehydrogenation; some methods furnished detailed information (individual compounds), others were short-cut methods. The results from the twelve participating laboratories were coordinated and published by Martin, Kurtz, *et al.* (6). The distillate had a boiling range of 72 to 124 C (by ASTM Method D 86),² and the saturates contained approximately 1 per cent C_5 , 36 per cent C_6 , 50 per cent C_7 , and 13 per cent C_8 hydrocarbons. The average value for naphthene content of the saturates portion was 56.3 per cent by volume. On the basis of this figure, then, an attempt at appraising the four charts was made. This time, however, the variation in the measured values for refractive index and density has been introduced. Thus Table IV shows the naphthene contents computed on the four charts by means of the physical property values obtained by Wood (Table VI of reference (2)) and in this laboratory as well as the difference from the average content obtained by the American Petroleum Inst. co-operators, employing several methods. There is a large spread in results due to the discrepancy between the values for physical properties obtained in the two laboratories, which indicates that the saturates portions isolated by them were not identical. This is an important phase of the problem and may explain the reason why cooperative testing of the refractivity intercept method has so far been inconclusive. For it is obvious that as a prerequisite for the attainment of an acceptable value for the total naph-

TABLE III.—COMPARISON OF REFRACTIVITY INTERCEPT METHOD WITH INFRARED SPECTROMETRY.

Source of Saturates Portion	Boiling Range, deg Cent	Difference ($n - d/2 - \text{IR Naphthene Contents, per Cent by Volume}$)			
		Wood's Special Charts ^a		Present Chart, ^b	ES-45a Chart, ^b
		Wide Range, 60 to 135 C	Narrow Range, 60 to 86 C	30 to 200 C	30 to 200 C
Straight Run Gasoline ^c	1 60 to 105	+3	..	-1	+1
	2 60 to 110	0	..	-3	-2
	3 60 to 110	+2	..	-2	+1
	4 60 to 110	+1	..	-3	-1
	5 60 to 110	0	..	-4	-2
Reformate ^d	6 100 to 140	+2	..	-1	-2
	1 60 to 110	+5	..	+4	-4
	2 60 to 110	+3	..	+1	-4
Straight Run Gasoline ^c	1 60 to 81	..	0	-1	+3
	2 60 to 81	..	+1	0	+4
	3 60 to 81	..	-2	-3	0
	4 60 to 81	..	+1	-1	-3
Reformate ^d	60 to 81	..	0	-2	-2

^a Wood's data from Tables IV and V of reference (2).^b Wood's refractivity intercept and density values applied to this chart.^c Naphthene contents, 50 to 60 per cent.^d Naphthene contents, 10 to 20 per cent.

TABLE IV.—NAPHTHENE ANALYSES OF THE SATURATED PORTION OF THE API COOPERATIVE SAMPLE BY THE REFRACTIVITY INTERCEPT METHOD USING VARIOUS CHARTS.

Assumed true naphthene content, 56.3 per cent by volume. W = Wood's charts or physical properties; A = Author's chart or physical properties.

Chart	Properties	Naphthenes, per cent by volume	Δ , per cent by volume
W Wide range (60 to 135 C).....	W	54.9	-1.4
	A	60.0	+3.7
W Narrow range (60 to 86 C).....	W	50.0	-6.3
	A	55.2	-1.1
A Full range (30 to 200 C).....	W	50.6	-5.7
	A	54.6	-1.7
ES-45a Full range (30 to 200 C)...	W	52.9	-3.4
	A	58.0	+1.7
Physical Properties	W		A
Refractivity intercept.....	1.0409		1.0403
Density.....	0.7231		0.7229

thene content, a saturated sample representative of the saturates actually present in the distillate must be secured. Although this can be achieved by elaborate large scale development chromatography, a routine method applicable to any straight run and cracked distillate remains to be established. In this case, Wood isolated the saturates by removal of the aromatics by acid absorption followed by neutralization, water-washing, and drying. We isolated them by displacement chromatography with a recovery of 99 per cent, thus obtaining a quite representative sample. It would seem from Table IV that the best results are obtained with our physical properties. On the basis of the latter values, three charts are about equally good, while the fourth, Wood's wide range chart, appears to be less applicable to the analysis of this distillate. This observation will be corroborated by a test, described below, to which more confidence can be accredited.

To our knowledge the only instance of an indisputable test for accuracy of the

charts is presented by the following analyses carried out in this laboratory. In the above reformer charge stock, all of the 25 commonly encountered saturated C_5 , C_6 , and C_7 hydrocarbons are present in ratios determined by the API cooperators. Fortunately all these compounds can now be purchased in high purity so that a synthetic mixture could be made to simulate the C_5 to C_6 saturates portion of the actual distillate. The boiling points of its components range from 36 to 104 C. The element of uncertainty ascribable to the technique of saturates isolation is of course eliminated in this test. The results are given in Table V.

The results of this test must be considered as decisive so that, as far as application to this type of sample is concerned, the charts can be listed in decreasing order of accuracy as follows:

1. Wood's narrow range chart
2. Author's full range chart
3. ES-45a full range chart
4. Wood's wide range chart

TABLE V.—NAPHTHENE ANALYSES OF A BLEND OF THE 25 COMMONLY ENCOUNTERED C_5 TO C_7 SATURATES BY THE REFRACTIVITY INTERCEPT METHOD.

Naphthene Content, 52.3 per cent by volume. Refractivity intercept, 1.0405(7); density, 0.7195(0).

Chart	Naphthenes, per cent by volume	Error, per cent by volume
Wood's wide range (60 to 135 C).....	56.6	+4.3
Wood's narrow range (60 to 86 C).....	51.8	-0.5
Author's full range (30 to 200 C).....	51.2	-1.1
ES-45a full range (30 to 200 C).....	54.1	+1.8

It should be borne in mind, however, that not only the charts but the whole refractivity intercept method can be fully evaluated only by application to representative samples of saturates isolated from numerous types of distillates, acceptable "true" naphthene contents of which have been determined by more elaborate means. To accomplish this, much work remains to be done.

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- (4) "Selected Values of Properties of Hydrocarbons and Related Compounds," American Petroleum Inst., Research Project 44, Carnegie Inst. of Technology, Pittsburgh, Pa.
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A Recording Torsion Testing Machine for Wire

By H. C. BURNETT

The recording system utilizes a spark and a heat-sensitive paper and is entirely frictionless, so that even small torque values may be accurately recorded

BECAUSE of the extensive use of small wires in helical extension or compression springs, the torsional properties of such materials are of major importance. In the course of an investigation conducted at the National Bureau of Standards under the sponsorship of the U.S. Army Ordnance Corps, Springfield Armory, a need was felt for a convenient means of determining these properties on wire less than 0.05 in. in diameter. Consequently a machine was designed and constructed which automatically records torque-twist measurements on small diameter wire. The recording method is entirely frictionless so that even small torque values are recorded accurately.

The following symbols are used:

- L = length over which twist is measured,
- d = wire diameter,
- θ = angle of twist (radians),
- γ = shear strain (radians),
- M = torsional moment,
- τ = shear stress, and
- G = shear modulus (11.5×10^6 psi for steel)

The general mechanical features of the torsion testing machine and automatic recording section are shown in Figs. 1 and 2. The framework consists of three horizontal plates clamped on vertical tubular members. The test specimen, *A*, is coupled in series with a larger diameter torque measuring wire, *B*. The bottom end of *B* is held in a coupling that is keyed to the bottom plate to prevent rotation and spring loaded to provide a small amount of tension to the two wires. The upper end of the test wire is rotated at an angular speed of about 100 deg per min by means of a motor, *C*, and reduction gear.

The rotation of the knife edge bar electrode, *D*, attached to the coupling between *A* and *B*, is proportional to the torque applied to *B*, as this wire is chosen large enough so that its elastic limit is not exceeded. A cylinder of heat sensitive recording paper, *E*, is attached to a disk mounted at the top of the test wire in such a way that the paper hangs between the moving bar electrode and

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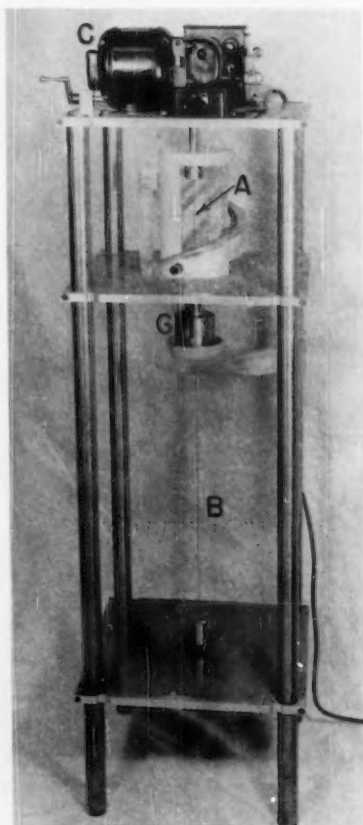


Fig. 1.—Torsion testing machine for small diameter wire.

A, specimen; *B*, torque measuring wire; *C*, motor and reduction gear assembly; *G*, damping cup.

the fixed knife edge electrode, *F*, without touching either. The movable and fixed electrodes *D* and *F* are designed with a knife edge to confine the origin and increase the intensity of the spark. An ignition coil and an interrupter (not shown in the figure) produce a spark between the electrodes which makes a dot on the paper. As torque is applied to the system by means of the motor, the difference in rotation of the recording paper and the bar electrode is a measure of the strain in the test wire. Rotation of the bar electrode is a measure of the torque

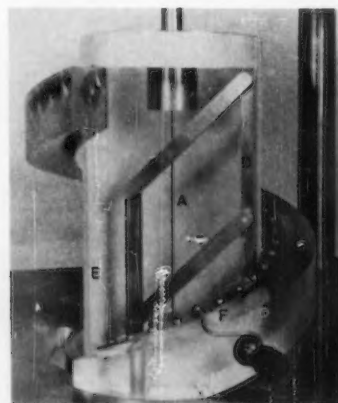


Fig. 2.—Recording section of torsion testing machine for small diameter wire.

A, test specimen; *D*, moving bar electrode; *E*, heat-sensitive recording paper, partially cut away to show specimen and moving electrode; *F* fixed helical electrode.

in the system, and is recorded by the vertical displacement along the helical fixed electrode. Sparks passing through the paper from the fixed electrode to the moving bar record the torque-twist relationship of the test wire. A damping cup, *G*, inverted in light oil eliminates oscillations.

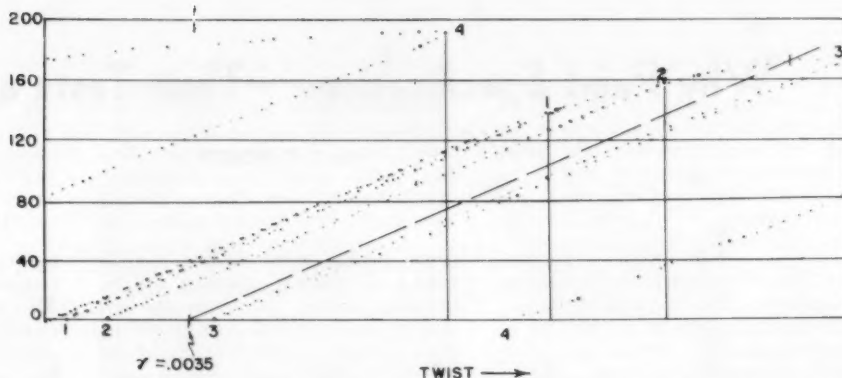
The effective gage length of the test wire is measured between right angle



H. C. BURNETT, Mechanical Metallurgy Section, National Bureau of Standards, Washington 25, D. C., has been engaged in research on the properties of wire and springs since 1951. His work has been concerned primarily with the factors influencing fatigue behavior and with new test methods for wire.

Fig. 3.—Photograph of the data obtained during a typical test to determine the load-permanent set characteristics of a sample of 0.039 in. diameter 1085 oil tempered wire.

The load is shown on the ordinate as nominal surface shear stress, 1000 psi.



bends near each end of the wire. The bent portions of the test wire are inserted into holes in the upper and middle grips so as to provide a positive means for the application of torque.

To simplify the strain measurements, the length of the test specimen was adjusted so that one 360-deg twist of the test wire would result in a convenient value of surface shear strain, γ . For example, when testing 0.039-in. diameter wire this value was adjusted to 0.02 as follows:

$$L = \frac{d\theta}{2\gamma}$$

$$L = \frac{2 \times 0.039 \times \pi}{2 \times 0.02} = 6.13 \text{ in.}$$

It has been estimated that 2.0 in.-lb would be the maximum torque required for testing 0.039 in. wire. From the relation

$$\tau = \frac{16M}{\pi d^3}$$

it was determined that 2.0 in.-lb would produce a maximum shear stress of 68,000 psi in 0.053 in. wire, so this size was considered suitable for the torque measuring wire. The length of this wire was then calculated from the following relation so that the 2.0 in.-lb of torque resulted in 270 deg of twist.

$$L = \frac{6G\pi d^4}{32M}$$

The recording sparks are produced by a motor-driven cam that closes and opens the primary circuit of an automobile ignition coil at 5 sec intervals. This timing provides sufficient space between the dots on the recording paper so that there is no tendency for a spark to go through the hole made by the previous discharge, rather than taking a direct path.

The torque-measuring wire and helix were calibrated by dead weight loading. The lower coupling was disconnected

from the bottom plate and torque was applied to the coupling by two fine wires wrapped around it. The wires passed over ball bearings mounted on opposite sides of the plate and supported pans in which balance weights were placed. A $\frac{3}{8}$ -in. diameter drill rod was substituted for the test wire, then the positions of both the bottom coupling and the vertical electrode were noted. A small increment of torque was applied by adding weights and the test section was then rotated by the motor until the free end of the torque-measuring wire returned to its original position. The location of the vertical electrode with respect to the helix was again recorded. The change in elevation was, therefore, a measure of the torque in the system. A series of similar tests were made with loads sufficient to traverse the entire height of the helix. A statistical analysis of the calibration data for both loading and unloading indicated a coefficient of variation in torque measurement of approximately 1.0 per cent.

Setting up the machine to run a test may be done quite rapidly. No special specimen preparation is necessary other than cutting and bending the ends 90 deg, with the desired test length between the bends. One end of the test wire, A, is then clamped in the fixture on the lower end of the drive shaft and the other end in the middle coupling that supports the moving electrode. A plastic tube of appropriate diameter, about which a sheet of heat-sensitive paper has been wrapped, is then placed in position around the test wire, middle coupling, and pointer so the paper may be slid into position and fixed to the flange attached to the upper coupling. After the paper is secured to the flange, the tube is removed and the torque-measuring wire secured to the middle coupling. Finally, the oil pan is placed in position so that the bottom of the damping cup is immersed in the oil.

The helix upon which the stationary electrode is secured is then rotated about the paper to the desired zero load position. The heat-sensitive paper may be sealed at appropriate torque and twist

values prior to the test and adjustments made during "setup" in positioning the paper and the helix to start the test recording at zero torque and twist positions. The drive motor and spark interrupter are then turned on and the test is in progress. During the test, sparks pass from the stationary helical electrode through the heat-sensitive paper to the moving vertical knife edge resulting in a line of dots recording the torque-twist data. A forward-neutral-reverse feature incorporated in the reduction gear assembly makes possible a variety of loading sequences.

Figure 3 is a typical example of a record obtained from a test to determine the torsional load-permanent set characteristics of a sample of 0.039-in. diameter 1085 oil tempered wire. For convenience the torque values have been sealed along the ordinate in terms of nominal surface shear stress. Twist increases to the right along the abscissa. During the test the specimen was alternately loaded and unloaded. The numbers 1 through 4 on the curve indicate the peak stress of successive cycles and the numbers along the base locate the total permanent set resulting from the corresponding cycle. It may be noted that both load-permanent set and twist-permanent set data are readily obtainable from the record sheet. The yield strength may also be determined as indicated by extension of the dashed line from an offset of $\gamma = 0.0035$. This value of offset has been used by others¹ for determining yield strength in torsion because it produces the same octahedral shear strain as a linear plastic strain of 0.2 per cent.

It is felt that this apparatus fills a gap in the presently available testing machines. The spark method of recording eliminates errors due to friction, so that small torques can be recorded accurately. The construction of the machine allows flexibility in the length of the test wire and the torque range of the calibrated wire to satisfy the requirements of the operator. In addition it has the advantages of low first cost and an absence of complicated accessories.

¹ R. D. Ollerman, E. T. Wessel, and F. G. Hull, "A Study of Factors Controlling Strength in the Torsion Test," *Transactions, Am. Soc. for Metals*, Vol. 46, pp. 87-99 (1954).

Why Paint Specifications—Their Tests and Controls

By C. F. PICKETT

THE WORD "specification" has different meanings to different individuals. The standard dictionary defines specification as (1) the art of specification, (2) a definite particularized and complete statement; full and definite account. The purpose of a specification is to define, particularize and completely state a condition of composition, shape, size, color, procedure, performance, or combination of these, with tolerance limits so that there is a common understanding between buyer and seller.

Why Paint Specifications

Should everyone buying paints use specifications? There are many cases where the use of specifications is not practiced. The user can depend on the integrity of the supplier not only to recommend the most suitable product but to maintain a uniform quality with each shipment. Most vendors in such cases will supply the necessary engineering service to obtain satisfactory results and will keep the user advised of new developments in the field. When such conditions exist, the buyers are also in the position of changing suppliers at their own discretion. This type of business arrangement does not work out where strictly competitive buying is involved, or where there are a number of suppliers; the reasons are self evident. Specifications are essential to large manufacturers, or those having multiple plants, to those industries where the paint or varnish product is essential for proper functions and performance of the end product, and especially to local, state, and Federal governments where competitive buying is customary.

Function of a Paint Specification

A paint specification should be a document which describes the product so well that the buyer obtains the desired quality and the vendor knows precisely the quality required. The function of the specification is to carry a complete message in so far as quality and performance are concerned. Well-prepared specifications will permit the buyer to have multiple suppliers of a

given quality paint, making possible mixing of products, stripping and re-finishing rejects, fewer solvents and uniform products from multiple plants. With these and several other advantages it is obvious that a specification is a useful item to a paint buyer. By the same token it provides fair competition to the seller and an opportunity to exercise ingenuity in formulations and manufacturing.

Types of Specifications

There are many types of specifications describing composition, performance, dimension, procedure, or combinations of these elements. Paint specifications frequently fall into the class of composition or performance. It is the opinion of the author that neither one alone is sufficient to completely describe a paint product. Since the very purpose or function of a specification is to describe a product so completely that both buyer and seller know what is wanted, all efforts should be made to do so.

Composition Specifications

If composition alone is used, there is bound to be misunderstanding. There is much art and science in compounding, blending, and processing a finished paint product. A paint formula is of limited value in inexperienced hands. In composition specifications the matter of analysis is a problem. Our analytical procedures are still lacking in many cases, especially after a complete paint has been processed. Examples are type and amount of phenol, certain types of dibasic acids, oils, amount of rosin, and amount of many synthetic and natural resins. Analysis of pigments is also limited by interfering reactions in certain areas, and in many cases the chemical analysis is not the critical measure of quality. Obviously then a strictly composition formula would certainly lead to an area of conflict because accurate analysis is not always possible and in many cases would not alone serve as a good criterion of quality.

Performance Specifications

How about performance alone? Those familiar with paint evaluation appreciate the difficulties and pitfalls. There are the usual tests of color, speed of drying, hardness, viscosity, opacity, adhesion, package stability, settling, water resistance, gasoline resistance,

salt fog box tests, accelerated weathering tests, and many other specialized tests. All of these are of much value in quality control of a given type product. However, in the final analysis the real test is the performance of the paint under actual service requirements. The paint industry is aware of the need for better methods of physical testing that can be correlated with actual service requirements. Many individuals and groups have spent much time on their study, and while much has been learned, frequently correlation between observers or laboratories leaves much to be desired. Even atmospheric exposures which may run for years are dependent on care of panel preparation. Further complication is encountered depending on the season the atmospheric exposure test is started plus the unpredictability of the elements themselves. Because of these many complications and the time involved in securing actual performance data, physical performance alone is not a suitable medium for a procurement specification.

Composition and Performance Specifications

Many paint formulators and users, based upon years of experience, can make a fair appraisal of the expected performance of a paint film from knowing the composition. If both composition and physical tests are required to a degree, it is possible to obtain a given type of product properly formu-



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lated, compounded, and processed. The composition should be sufficiently described to permit blending of lots, eliminate difficulties due to wrong solvents, permit stripping and repainting of rejects and touch-ups. The physical tests should insure the use of proper grades of components plus proper compounding and processing. Some view a specification based upon both composition and performance as being rigid. It is believed that sufficient parameters of both composition and performance should be included in a specification so that the type, quality, and performance are assured. Such a specification invariably works to the advantage of both the buyer and seller. Beware of such phrases as "wide scope in composition is allowed," or "manufactured in accordance with standard or acceptable commercial practice," or "applied in a highly workmanlike manner," or "high quality durable product," and many other hackneyed phrases. Such phrases are not factual, and lend themselves to many interpretations and almost always to an area of conflict and misunderstanding. How much better it is to describe composition, quantity, and physical properties that can be measured and, when integrated, describe a product of known performance.

Complete Specifications

The physical putting together of a specification is one of mechanics. There are several acceptable formats. A needed ingredient is a knowledge of paints—both composition and performance. With this knowledge it is possible to select those parameters of composition and performance to completely describe a paint product and eliminate the loopholes. The loopholes are disconcerting to the buyer and an aggravation to the seller who is competitively trying to meet both the spirit and letter of the specification. It is readily apparent that the many tests of composition, physical properties, and procedure are the essential components needed to write a specification. It is also self evident that the tests used, in the hands of competent personnel, should be reproducible, to within stated limits of accuracy.

ASTM Committee D-1

ASTM Committee D-1 on Paint, Varnish, Lacquer, and Related Products has been in existence for over fifty years. Its membership consists of in-

dividuals having a wide divergence of interests including consumers, paint manufacturers, raw material manufacturers and suppliers, association members, university faculty members, and representatives of local, state, and Federal governments. However, the one common interest is to cooperate in developing some new specification or method of test. The working of the various committees sometimes appears slow, but the end product after undergoing committee scrutiny and cooperative testing is the sort of test one can include in a specification with a high degree of assurance. The interests of Committee D-1 are very diverse and cover chemical methods of analysis, physical tests, test methods and procedures. One might wonder if all test procedures have not already been worked out. Unfortunately they have not. The need for new test methods will not stop until the paint industry ceases to progress. If one can predict the future from a look into the past it is very obvious that great strides will be made in the future creating more needs for new and different test methods.

Subcommittee XI of Committee D-1

Subcommittee XI, on Resins, of which the author is now chairman, was founded some eight years ago under the chairmanship of the deceased Mr. Matello. It was believed that the field of resins was of sufficient importance to establish a separate subcommittee. Nine methods have been added, namely, D 563 - 52¹ Test for Phthalic Anhydride Content of Alkyd Resins and Resin Solutions, D 1013 - 52¹ Test for Total Nitrogen in Resins for Surface Coatings, D 1156 - 52¹ Test for Total Chlorine in Polyvinyl Chloride Polymers and Copolymers Used for Surface Coatings, DD 1198 - 55¹ Test for Solvent Tolerance of Amine Resins, D 1259 - 56² Test for Nonvolatile Content of Resin Solutions, D 1306 - 56² Test for Phthalic Anhydride Content of Alkyd Resins and Esters Containing Other Dibasic Acids (Gravimetric), D 1307 - 56² Test for Phthalic Anhydride Content of Alkyd Resins and Esters Containing Other Dibasic Acids (Spectrophotometric), D 1312 - 56² Test for Apparent Free Phenols in Synthetic Phenolic Resins or Solutions Used for Coating Purposes, D 1257 - 55¹ Specifications for High Gravity Glycerine, D 1258 - 55¹ Methods of Sampling and Testing High Gravity Glycerine. Indications are that several additional methods will soon be submitted to letter ballot for acceptance as tentative methods. These include methods for the determination of unsaponifiables, the determination of fatty

acids, the determination of polyhydric alcohols, namely pentaerythritol, glycerol and ethylene glycol, and the determination of polyvinyl alcohol, polyvinyl acetate and total butyraldehyde. Projected work includes the determination of phthalic anhydride in the presence of nitrocellulose, polystyrene in styrenated alkyds, isophthalic, teraphthalic, and orthophthalic present in the same resin, benzoic acid, melamine, urea, and analytical methods for epoxy resins. This list is not all inclusive but does indicate the scope of interest and work. The delay in accomplishing the work at a faster rate is simply lack of working people. All of the methods listed, those nearing completion and those which are projected furnish some of the components necessary to prepare adequate paint specifications.

Changing Requirements

Staying within the scope of Subcommittee XI, it is apparent that in the short space of eight years the industry has changed to a degree requiring new or improved methods. The determination of phthalic anhydride is a good example. The Kappelmeier method is not adequate in the presence of other interfering dibasic acids. A newcomer, isophthalic acid, presents another analytical problem. The advent of styrenated alkyds presented a problem both in the analysis of phthalic anhydride, and also the analysis of polystyrene. The analysis of vinyl resins is a relatively new requirement. Epoxy resins are already widely used and needing a method of analysis. These are but a few of more or less current changes that require additional work. Multiply the changes in other components that are covered by other subcommittees of Committee D-1 and it becomes apparent that much is going on in Committee D-1 that is of interest to all connected with the paint industry.

Origin of Test Methods

To many unfamiliar with ASTM's operation, the question arises as to where the ideas for the various test methods originate. There is no fixed source of ideas or starting point. Once agreement for the need of a test method has been reached, the next step is usually arrived at the working group level and may involve a literature search, the suggestion of a method already published in some technical journal, and very frequently a method used in some member's laboratory. The proposed method is presented and discussed by the membership. Nearly always, someone has had some experience with the method in question. When several

¹ 1955 Book of ASTM Standards, Part 4.

² 1956 Supplement to Book of ASTM Standards, Part 4.

people have had some previous experience, difficulties in procedure or areas requiring special care or technique are usually brought out. Usually a proposed procedure will be drawn up incorporating the many refinements or cautions to be observed. This is usually followed by a "round robin" where several laboratories try the method. Results are then compiled, studied, and reconciled. The method is frequently further refined by more carefully describing operation procedures and condi-

tions and again resubmitted to a round robin test. When the working group is satisfied with the reproducibility and accuracy the method is then submitted to the subcommittee for letter ballot. If the method passes the subcommittee it is then submitted to Committee D-1 for letter ballot. The idea for a test method may come from many sources but before it is accepted as a tentative method it has had to undergo laboratory test with several collaborators and then subjected to the scrutiny of a large membership.

Conclusion

In conclusion it may be stated that paint specifications are a useful item to paint users. Paint specifications should be so complete that they leave no area of conflict or argument. Paint specifications require test methods and procedures as essential components. The test methods must be highly reproducible and tried. The ASTM is actively engaged in developing new test methods and procedures needed for paint specifications.

Element 41—Historical Priority or International Unity?

COLUMBIUM or niobium (which do you use?) are the names commonly associated with element 41. The American Chemical Society has adopted "niobium"; American metallurgists still prefer "columbium," whose practice is followed by the ASTM. The chemists are using niobium in the interests of internal unity; the metallurgists are using columbium because of the prior use of that name and because development of "columbium" as an important commercial metal has resulted primarily from work done by the metallurgists in America.

In 1949 the Commission on the Nomenclature of Inorganic Chemistry of the International Union of Pure and Applied Chemistry recommended to the various national committees that element 41 be called "niobium." The Nomenclature Committee of the National Research Council (the official U. S. representative in the IUPAC) agreed to this recommendation and in May 1951, the Nomenclature, Spelling and Pronunciation Committee of the American Chemical Society also agreed.

Another recommendation in 1949 by the Commission of the IUPAC was the use of "wolfram" for element 74. However, at another meeting of the IUPAC Commission in 1950 the recommendation was changed so that element 74 could be called either "wolfram" or "tungsten." This change was made after a decided preference for "tungsten" was expressed in England and America. This was not true for the term "columbium" which is common to America only.

In the interests of international standardization and utility, therefore, the American chemists agreed to use "niobium" in place of "columbium," although they recognized that "columbium" had a better claim historically. Having opposed "wolfram" on the basis

of usage rather than historical claim, it seemed best to concede similar reasons held for "niobium" by most Europeans. The French made a similar concession in giving up "gluconium" for "beryllium."

In giving the metallurgists' claim for historical preference it is only necessary to quote an abstract from a historical note by J. Lawrence Smith, printed in May, 1877, in the *American Journal of Science*.

It is the common practice of all American chemists and mineralogists to speak of the metal which is called "Niobium" by English and continental chemists, as "Columbium." This is eminently just, since the metal was discovered and well defined, and named columbium, forty-five years before the name niobium was given to it. The change of name was caused by a double mistake, in no way connected with the original observations in 1801 by Prof. Hatchett of England.

These mistakes arose as follows:—In the first place, Ekeberg discovered in 1802 a supposed new metal which he called tantalum, but which a short time afterward was regarded as identical with columbium; and for forty-five years tantalum and columbium were synonymous terms in all works on chemistry, although Wollaston suspended their dissimilarity; secondly, when H. Rose made his well-known exhaustive researches on the columbite of Bodenmais, he showed that this mineral contained not one but two metallic acids; one of these was tantalum, and the other he supposed to be a new metal which he named niobium.*

Subsequent examination, however, convinced Rose (and his conclusions have been confirmed by others who have repeated his experiments), that the two metallic acids obtained from the Bodenmais columbite were really the original columbic acid of Hatchett, discovered in 1801, and the tantalic acid discovered by Ekeberg in 1802. Instead, however, of calling the first mentioned acid niobic acid, its original name should have been left to it. The

* H. Rose still supposing that this tantalum was the same as what was known equally well as columbium.

result of Rose's researches was in fact simply the demonstration of the actual difference of columbium and tantalum; for Hatchett's discovery was clear, precise, and well made out, and has never been controverted.

This being a correct summary of the history of the composition of the columbium minerals, it is but right, just, and in accordance with chemical and mineralogical precedence, that the name given by the discoverer should replace that of niobium, which originated forty-five years later.

A point of less importance, but worthy of some consideration, is, that this element derived its name from the country in which it was discovered, it being the first and up to the present time, the only element discovered in that part of America usually named Columbia.

It does not appear that there is any solution to this impasse between American technicians, and that metallurgical literature will, for the most part, continue to use "columbium" whereas chemical literature will use "niobium." In view of the fact that the International Commission has approved the use of either "tungsten" or "wolfram" for element 74, perhaps the use of either "columbium" or "niobium" could be approved.

25 Years for Metals Research Lab at Carnegie Tech

THE Metals Research Laboratory of Carnegie Institute of Technology observed its twenty-fifth anniversary with a full-day reunion program in Pittsburgh on October 24. The Laboratory is a special research institute associated with the Department of Metallurgical Engineering in the College of Engineering and Science.

Visiting the Carnegie campus to honor the Laboratory and its director, Robert F. Mehl, were 300 graduates of Tech's Metallurgical Engineering Department, former Carnegie faculty members, and friends from industry.

Federal Government Standards Index Changes

The General Services Administration of the Federal Supply Service is charged with the responsibility for establishing specifications to be used by the Federal Government for procurement of materials and supplies. The GSA issues an annual Index of Initiation of Federal Specifications Projects as of March 1, and monthly supplements.

The items listed below appeared in Supplement No. 8 for the month of October 1957.

INITIATIONS:

Title	Type of Action	Symbol or Number	FSC Code	FSSC Class	Assigned Agency and Preparing Activity
Aggregate; (for) Portland-Cement Concrete	Rev.	SS-A-281b	COM-BPR
Aluminum Alloy Sand Castings	Am. 1	QQ-A-601b	GSA-FSS
Bottle, Stopper, Ground Glass Cap	New	DD-B-00595	DOD-MMSA
Bottles, Weighing (With Stoppers)	Am. 1	DD-B-620a	6640	..	GSA-FSS
Boxes, Fiber	Am. 1	PPP-B-636	8115	..	DOD-Army-QMC
Cloth, Cotton, Seersucker	New	CCC-C-448	8305	83	DOD-Army-QMC
Crucibles and Covers, Porcelain	Rev.	DD-C-00686b (HEW-PHS)	6640	..	HEW-PHS
Distilling Apparatus with Graham Condenser	Rev.	GG-D-00461b (HEW-PHS)	6640	..	HEW-PHS
Flasks, Laboratory	Am. 1	DD-F-250	6640	..	GSA-FSS
Funnels, Filtering, Glass, Buechner Type	Rev.	DD-F-00776b (HEW-PHS)	6640	..	HEW-PHS
Funnels, Filtering, Porcelain, Buechner	Rev.	DD-F-00786b (HEW-PHS)	6640	..	HEW-PHS
Gauze; Wire (For Laboratory Use)	Rev.	RR-G-00123a (HEW-PHS)	HEW-PHS
Insulation, Blanket, Thermal-Acoustical, and Insulation, Thermal; Vegetable or Wood Fiber	Am. 1	HH-I-515	DOD
Phosphor Bronze Bars, Plates, Rods, Sheets, Strips, Flat Wire, and Structural & Special Shaped Sections	Rev.	QQ-P-330	9525 9530 9535 9540	..	DOD-Navy-Ships
Plastic Compounds, Molding & Extrusion, Polyethylene	Am. 1	L-P-590	9330	42	DOD-Navy-Ships
Plastic Film, Polyethylene, Thin Gauge	New	L-P-378	9330 8135	..	DOD-Army-QMC
Plywood, Flat Panel	Rev.	NN-P-530	DOD-Army-CE
Roofing Felt, Glass Fiber, Asphalt Saturated, Uncoated (For Flashing and Roofing)	New	SS-R-00620 (GSA-FSS)	GSA-FSS
Sealing Compound, Fuel Resistant, Hot Applied Concrete Paving	Rev.	SS-S-167b SS-S-00167a (DOD-Army-CE)	DOD-Army-CE
Sealing Compound, Two Components, Jet Fuel Resistant, Cold Applied, Concrete Paving	Rev.	SS-S-00170a (DOD-Army-CE)	5610	..	DOD-Army-CE
Tape, Pressure-Sensitive Adhesive (Cellophane & Cellulose Acetate)	New	L-T-90	7510 8135	..	GSA-FSS
Tape, Pressure-Sensitive Adhesive, Polyester Film	New	L-T-100 and L-T-00100 (GSA-FSS)	7510 8135	..	GSA-FSS
Tape, Pressure-Sensitive Adhesive, Vinyl Plastic Film	Rev.	PPP-T-66	7510 8135	..	GSA-FSS
Washer, Pipette	Rev.	GG-W-00105b (HEW-PHS)	6640	..	HEW-PHS

TITLE AND SYMBOL CHANGES:

Title	Type of Action	Symbol or Number	Former Title or Symbol
Boxes, Fiberboard, Corrugated, Triple Wall	Rev.	PPP-B-640 PPP-B-00640a (USAF)	Boxes, Folding, Fiberboard, Corrugated, Triple Wall

WITHDRAWALS:

Title	Type of Action	Symbol or Number	Assigned Agency and Preparing Activity	Reason for Withdrawal
Molding Plastic, Polyvinyl Chloride Rigid, Rods and Tubes	New	L-M-540	DOD-Army-Ord	Discontinued at request of DOD
Polyethylene Fabricated Parts	New	L-P-570	DOD-Army-Ord	Discontinued at request of DOD

REASSIGNED PROJECTS:

Title	Type of Action	Symbol or Number	FSC Code	FSSC Class	Technical Former Agency Withdrawn from	Committee or Reassigned to
Blankets, Other than all wool	Rev.	DDD-B-421c DDD-B-00421c (DOD-QMC)	7210	72	JUS-FPI	DOD-Army-QMC

PROMULGATIONS:

Title	Type of Action	Symbol or Number
Boxes, Wood, Nailed and Lock-Corner	Am. 1	PPP-B-621
Brass, Leaded and Non-Leaded; Plate, Rolled Bar, Sheet and Strip (Superseding FS QQ-B-613 (In Part))	Rev.	QQ-B-513a
Bronze, Manganese and Aluminum-Manganese, Castings (Superseding FS QQ-B-726c)	Rev.	QQ-B-726d
Cleaning Compound, Soap Abrasive, Paste, for Painted Surfaces (Superseding FS P-C-565)	New	P-C-442
Felt, Sheet, Wool, Pressed	Am. 1	C-F-206a
Hydrometers, Graduated Scale (For General Laboratory Use)	New	GG-H-925
Molding Plastic, Polystyrene (Superseding Fed. Spec. L-P-416 (In Part))	New	L-M-520
Molding Plastic, Polystyrene, Modified	New	L-M-525
Soap, Laundry, Bar (Superseding IFS P-S-00591d (GSA-FSS) & FS P-S-591c)	Rev.	F-S-591e
Steel Strapping, Round (Bare and Zinc-Coated) (Superseding FS QQ-S-790a)	Rev.	QQ-S-790b
Tube, Magnesium-Alloy, Extruded (Superseding FS WW-T-825)	Rev.	WW-T-825a

INTERIM FEDERAL SPECIFICATIONS ISSUED:

Title	Type of Action	Symbol or Number
Cloth, Cotton, Broadcloth, Mercerized	New	CCC-C-00437(GSA-FSS)
Methyl Isobutyl Ketone (For Use in Organic Coatings)	New	TT-M-00268a(GSA-FSS)
Paperboard, Wrapping (With Cushioning Value)	New	PPP-P-00291(COM-NBS)
Tape, Pressure-Sensitive, Adhesive (Cellophane & Cellulose Acetate)	Rev.	L-T-0090a(GSA-FSS)
Tape: Pressure-Sensitive Adhesive, Vinyl Plastic Film	Rev.	PPP-T-0066a(GSA-FSS)

CANCELLATIONS:

Title	Symbol or Number	Reason for Cancellation
Cadmium; Anodes	QQ-C-61	Superseded by Fed. Spec. QQ-A-671
Glassware, Laboratory; Methods for Sampling & Testing	DD-G-626a	" " " Test. M. No. (626)
Plastics; Cellulose Acetate, Molded	L-P-344	" " " Spec. L-M-505

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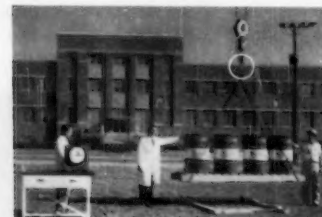
It polymerizes



1. At high noon we put one drop of something on the end of a clean 2" steel rod.



2. We butt it against another such clean rod, pressing firmly with the fingers.

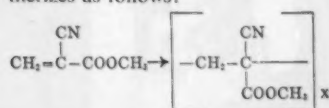


3. By 12:15, our single drop has formed a bond of considerable tensile strength.



4. And by 12:30 it is even stronger. (Tests show it to be over 5,000 psi.)

The liquid in the dropper is a chemical invention we call *Eastman 910 Adhesive*. Its major ingredient is a thin, watery liquid which polymerizes as follows:



With very few exceptions, polyethylene and silicone grease among them, it sticks all materials together—like and unlike, organic and inorganic, metallic and non-metallic. Temperatures above 100 C or continuous high humidity above 80 C eventually spoil the bond.

For a one-ounce sample of Eastman 910 Adhesive send \$5 to Eastman Chemical Products, Inc., Kingsport, Tennessee (Subsidiary of Eastman Kodak Company). Part of the reason for the price is the high attrition rate of the production equipment. The manufacture of this product can suddenly turn into the most awful mess you ever saw in your whole life. Nevertheless, the cost should decline some when the production volume rises.

Brute force

We make a kind of photographic paper on which you can write with a light beam and see the writing immediately, with no chemical processing whatsoever. Paper which requires no processing is of itself no news.* There may be a little news in that at least two major manufacturers of moving-mirror oscillographs now offer recording instruments based on what we call *Kodak Linagraph Direct Print Paper*. If they want to call it something else, we love them none the less.

In giving up photographic development, one gives up for the sake of dry fingers an energy amplification factor of enormous power—a main source, indeed, of the power photography has had in the world of technology to date. Nevertheless, we agree that dry fingers are nicer than wet fingers. We bow low to the optical design ingenuity that has contrived cool and quiet little boxes in which most satisfactory traces are put down through brute force of u-v radiation. One company claims frequencies to 2,000; the other, to "above 3,000," with trace velocities "above 30,000 inches per second."

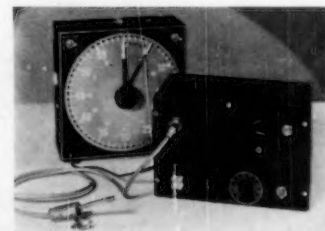
At low recording and writing speeds, focused energy from a high-intensity Hg-vapor ultraviolet point source is sufficient for a legible record. Where it isn't, use is made

* For better than 60 years we have made studio proof paper. On it many portrait photographers submit those brownish-purple proofs from which the customers select the poses they like. The proofs are to be returned. Cheats, who instead stick them up on their boudoir mirrors, find they don't last very long in the light of day. That's the idea with studio proof paper—no developing and no fixing.

of a post-exposure to some 60 foot-candles from a fluorescent lamp. If speed is defined as the reciprocal of the energy required to produce a given recognizable increment of trace density over background density, the post-exposure raises the speed several hundred times. The trace comes out blue on a buff background. As with most miracles, the miracle of *Kodak Linagraph Direct Print Paper* becomes a little less miraculous upon quantitative study. The total energy delivered to the paper in the writing and the post-exposure is about the same as a conventional photographic enlarging paper. It's just that the energy requirement can be supplied in two separate doses.

If interested in this type of oscillography, watch for pertinent ads in the technical press and alert your purchasing agent to send in the next man who comes around with one of these instruments. If you have other ideas for this type of paper, Eastman Kodak Company, Graphic Reproduction Division, Rochester 4, N. Y., will try to be helpful by arranging with a local dealer to sell you some of it for experimentation.

Arrhenius's clock



Time, as told by this clock, elapses at a rate that is temperature-dependent in the same way as the rate of a chemical reaction is temperature-dependent. Sensing is done by the thermistor probe, which goes into the reaction vessel. It's handy in a photographic lab because by time compensation it tames a ± 4 F temperature variation to the equivalent of ± 0.2 F control. Arrhenius, who wrote the equation but wasn't as hipped on photography as we are, would have been pleased.

Though we don't manufacture these for sale, we'll be glad to furnish a reprint that tells how we made ours. Drop a note to Eastman Kodak Company, Special Sensitized Goods Division, Rochester 4, N. Y.

Price quoted is subject to change without notice.

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This is another advertisement where Eastman Kodak Company probes at random for mutual interests and occasionally a little revenue from those whose work has something to do with science

PERSONALS...

News items concerning the activities of our members will be welcomed for inclusion in this column

B. B. Bachman, whose career has almost spanned the automobile age, is going into semiretirement at The Autocar Division, The White Motor Co., Exton, Pa. Mr. Bachman, who joined The Autocar Co. as a draftsman in 1905, will continue, however, as director of engineering on a part-time basis.

Lester Benoit has become secretary-treasurer of the Pipe Fittings Manufacturers Assn. Until recently he was general secretary of the Manufacturers Standardization Society of the Valve and Fittings Industry.

W. C. Bowden, technical director, Ledoux and Co., Teaneck, N. J., serves as treasurer of The Technical Societies Council of New Jersey.

Ira S. Boydston, formerly with Petrolite Corp., is now with Chemical Construction Corp., Houston, Tex.

Achilles W. Coutris, formerly with Moran, Proctor, Mueser & Rutledge, New York City, is now consulting engineer, Société Technique pour L'Utilisation de la Précontrainte, Paris, France.

Théodore S. Du Mond has been appointed to the American Society for Metals staff to serve as editor-in-chief of the educational courses prepared by the Metals Engineering Institute. In addition he will be secretary of the ASM Special Engineering Program Committee. Prior to joining the ASM staff, Mr. Du Mond was editor-in-chief of *Materials and Methods*.

Kenneth Forrest, formerly chief engineer, Soiltest, Inc., Chicago, Ill., is now president, Dynamics, Inc., Bensenville, Ill.

Miro A. Grottger, until recently chief chemist, Walker Brothers of Conshohocken (Pa.) is now vice-president and

technical director, Industrial Finishes Co., Inc., Philadelphia, Pa.

Pieter Honig, formerly head, Technical Research Div., West Indies Sugar Corp., New York City, is now consultant to agricultural industries, Elmsford, N. Y.

Richard B. Kropf has been appointed supervisor of development activities in the automotive industry, International Nickel Co., with his headquarters continuing in Detroit. He has been with the company since 1945.

Max Henry Laden has been given a foreign assignment by Swift & Co. as technical adviser in a new adhesive plant of Swift & Co., Ltd., Ashford, England. Mr. Laden has been representing his company on ASTM Committee D-14 on Adhesives.

Raymond W. Meyer, formerly with L.O.F. Glass Fibers Co., Houston, Tex., is now superintendent, Reinforced Plastics Dept., Gale Products Div., Outboard Marine Corp., Galesburg, Ill.

Leon J. Printz has accepted a position as service sales engineer, Republic Steel Corp., Detroit, Mich. He was previously associated with Amplex Mfg. Co., Division of Chrysler Corp., in the same city.



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FOR FURTHER INFORMATION CIRCLE 761 ON READER SERVICE CARD

Ole Singstad, tunnel engineer, and partner, Singstad & Baillie, New York City, received the 1957 award of the New York Chapter of the New York State Society of Professional Engineers for distinguished engineering services.

Warren M. Spear, staff metallurgist, Worthington Corp., Harrison, N. J., has been elected chairman of the AFS Gray Iron Division on Effect of Temperature on Properties of Cast Iron.

John F. Thompson, chairman of the board of The International Nickel Co. of Canada, Ltd., will be the recipient of the 1958 Charles F. Rand Memorial Medal of the American Institute of Mining, Metallurgical, and Petroleum Engineers. He is recognized "for leadership of a mining and metallurgical enterprise which has contributed to free world strength; for administrative skills which have furthered the progress of the nickel industry; for his vision in stimulating scientific research; for demonstrating the value of a technical education combined with broad interests and human understanding." Presentation will take place on February 19 at the AIME annual banquet in New York City. Dr. Thompson is a 50-year ASTM member.

Maxwell M. Upson recently completed his 50th year as an officer and top executive of the Raymond Concrete Pile Co., New York City. As chairman of the board for his company, at 81 he still is intensely active in the fields of business, engineering, and finance, as well as in education and religion.

James P. Wallace has accepted a position as director of testing, H. C. Nutting Co., Miami, Fla. He formerly was with the Pittsburgh Testing Laboratory, Newburgh, Ind.

Robert V. Warrick has been appointed executive secretary of the Manufacturers Standardization Society of the Valve and Fittings Industry. He was formerly assistant utilization engineer, American Gas Assn.

Paul Latimer Weir, formerly with Potomac Electric Power Co., is now general engineer, Architect of the Capitol, U. S. Capitol, Washington, D. C.

N. G. Zoldners has been named senior research engineer, Constructions Materials Section, Industrial Minerals Div., Department of Mines and Technical Surveys, Ottawa, Canada. Until recently he was with Mount Royal Paving and Supplies, Ltd., in Montreal.

C. E. Davies, ASME, Secretary Retires; O. B. Schier II, Successor

Clarence E. Davies, for 23 years with the American Society of Mechanical Engineers, retired on December 2 but will continue in service to the engineering profession as coordinator for the United Engineering Center.

O. B. Schier II, formerly assistant and later deputy secretary, succeeds Mr. Davies as ASME secretary.

A graduate of Rensselaer, which school granted him an honorary doctor's degree, Mr. Davies had been affiliated with the ASME Staff for 38 years. In this capacity he was brought into contact with ASTM on many occasions and helped develop and maintain very cordial relations between the two organizations. He rendered notable service to the Defense De-

partment in World War II and has been a leader in the Engineers Joint Council.

Mr. Schier, a Lehigh graduate, has been with the ASME for some ten years.

At the 1957 ASME Annual Dinner, Mr. Davies received several gifts from his friends and associates which included a beautiful book of testimonials. His office as coordinator for the new Engineering Societies Building will continue at 33 W. 39th St., New York, N. Y.

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All Government certificates mentioned (hardness standards; diamond indentors; optical load indicators) are issued for our account by the Materials Testing Institute, Federal Republic of Germany.

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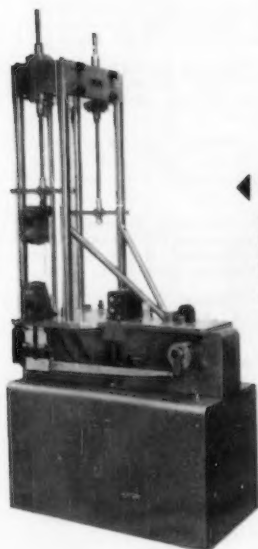
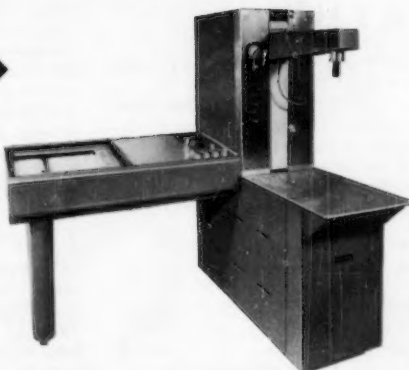
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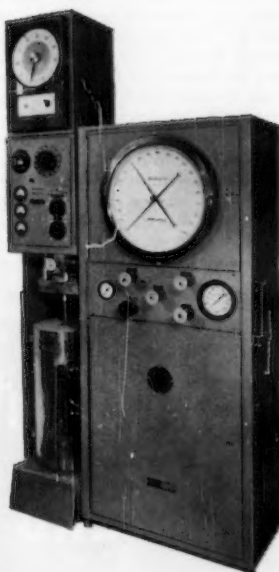
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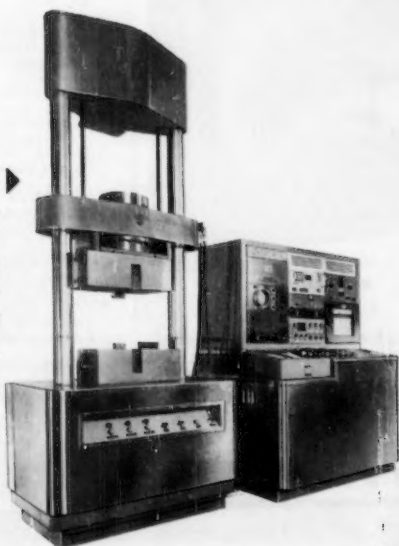


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with instrumentation for
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Model RL-24
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Load Capacity:
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20:1 lever ratio



FOR FURTHER INFORMATION CIRCLE 763 ON READER SERVICE CARD

DEATHS...

Johanna Busse Allen, National Bureau of Standards, Washington, D. C. A very loyal member of Subcommittee 17 on Thermometers of Committee E-1 on Methods of Testing since 1939, Mrs. Allen's respected advice on matters relating to thermometers and her friendly presence will be greatly missed.

William C. Du Comb, president, W. C. Du Comb Co., Inc., Detroit, Mich. (Nov. 22, 1957). Affiliated since 1902, Mr. Du Comb received his ASTM 50-year Membership Certificate at the 1952 Annual Meeting in New York City. A member of the staff of the Riehle Bros. Testing Machine Co. for many years, and later Detroit district manager for American Chemical Paint Co., of Ambler, Pa., Mr. Du Comb in 1915 founded his own firm which specializes in the supply and engineering of ball and roller bearings and power transmission equipment. A loyal member of the Society for 55 years and very active in Detroit District matters, Mr. Du Comb through his friendly, dynamic personality and widespread acquaintances, interested many people in the work of ASTM and in attendance at meetings.

P. T. Naudet, director of technical research for Consumers Cooperative Assn.,

died at his home in Kansas City, Mo., Dec. 11, 1957. Mr. Naudet had an important part in the development of CO-OP Multi-Purpose Grease and other products manufactured by CCA. A member of ASTM since 1954, Mr. Naudet had been elected a member of Committee D-2 on Petroleum Products and Lubricants in 1957, and was serving on Technical Committees B on Lubricating Oils, F on Diesel Fuels, G on Lubricating Grease, and L on Tractor Fuels.

Thorsten Y. Olsen, chairman of the board of the Tinius Olsen Testing Machine Co., Willow Grove, Pa., died Dec. 11, 1957, at the age of 78. Mr. Olsen had been connected since 1903 with the firm founded by his father, Tinius Olsen, in 1879, serving as president from 1929 till 1955 when he relinquished the presidency to his son Tinius Olsen II. Widely known throughout industry, Mr. Olsen was the author of numerous articles on the art of testing materials and balancing rotating parts. He also was known for his many civic activities and for his interest in his father's native land, Norway. In recognition of his contributions to the advancement of technical training in Norway he was knighted into the Order of St. Olaf by King Haakon in 1935, one of the highest civilian honors granted by the king. Mr. Olsen was a personal member of ASTM for 26 years.

Through corporate and individual mem-

berships, and active participation in technical committee work, the Olsen family has been a part of ASTM from the Society's inception. Tinius Olsen, the founder of this testing company, one of the largest in the industry, held membership from 1898 till his death in 1932.

Tinius Olsen II (son of Thorsten) has been an active member since 1935.

OTHER SOCIETIES' EVENTS

February 2-7—**American Institute of Electrical Engineers**, Winter General Meeting, Hotel Statler, New York, N. Y.

February 3-4—**Instrument Society of America**, National Chemical and Petroleum Instrumentation Conference, Hotel du Pont, Wilmington, Del.

February 4-6—**Society of the Plastics Industry**, Reinforced Plastics Div. Conference, Edgewater Beach Hotel, Chicago, Ill.

February 10-13—**National Sand and Gravel and National Ready Mixed Concrete Assns.** Joint Convention, Conrad Hilton Hotel, Chicago, Ill.

February 13-15—**National Society of Professional Engineers**, Spring Meeting, Michigan State University, East Lansing, Mich.

February 16-20—**American Institute of Mining, Metallurgical and Petroleum Engineers**, Annual Meeting, Hotels Statler and Sheraton-McAlpin, New York, N. Y.

February 24-26—**National Concrete Products Assn.**, 9th Annual Convention, Seigniory Club, Montebello, Quebec.

February 24-28—**American Society of Civil Engineers**, National Convention, Hotel Sherman, Chicago, Ill.

February 25-26—**Society of American Military Engineers**, Midwestern Regional Meeting, Conrad Hilton Hotel, Chicago, Ill.

February 25-27—**American Concrete Institute**, 54th Annual Meeting, Morrison Hotel, Chicago, Ill.

March 11-13—**American Railway Engineering Assn.**, Annual Meeting, Sherman Hotel, Chicago.

March 17-21—**National Association of Corrosion Engineers**, Civic Auditorium, San Francisco, Calif.

March 17-21—**Nuclear Congress**, Coordinated by Engineers Joint Council International Amphitheater, Chicago, Ill.

April 13-18—**American Chemical Society**, Spring Meeting, San Francisco, Calif.

April 14-18—**American Welding Society, Inc.**, Annual Meeting and 6th Welding Show, Hotel Statler, St. Louis, Mo.

April 20-25—**American Water Works Assn.**, Dallas, Tex.

April 20-26—**Society of Motion Picture and Television Engineers**, Ambassador Hotel, Los Angeles, Calif.

April 21-23—**Building Research Institute**, 7th Annual Meeting, Shoreham Hotel, Washington, D. C.

April 21-23—**American Oil Chemists Society**, Spring Meeting, Memphis, Tenn.

April 27-May 1—**The Electrochemical Society**, Statler Hotel, New York, N. Y.

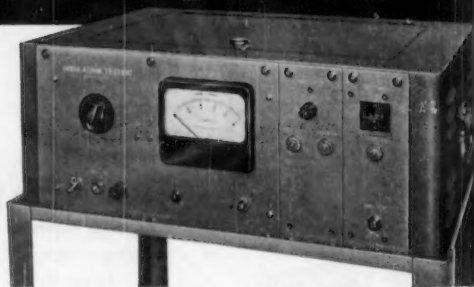
April 28—**The Textile Institute**, (Great Britain), Annual General Meeting, Nottingham, England

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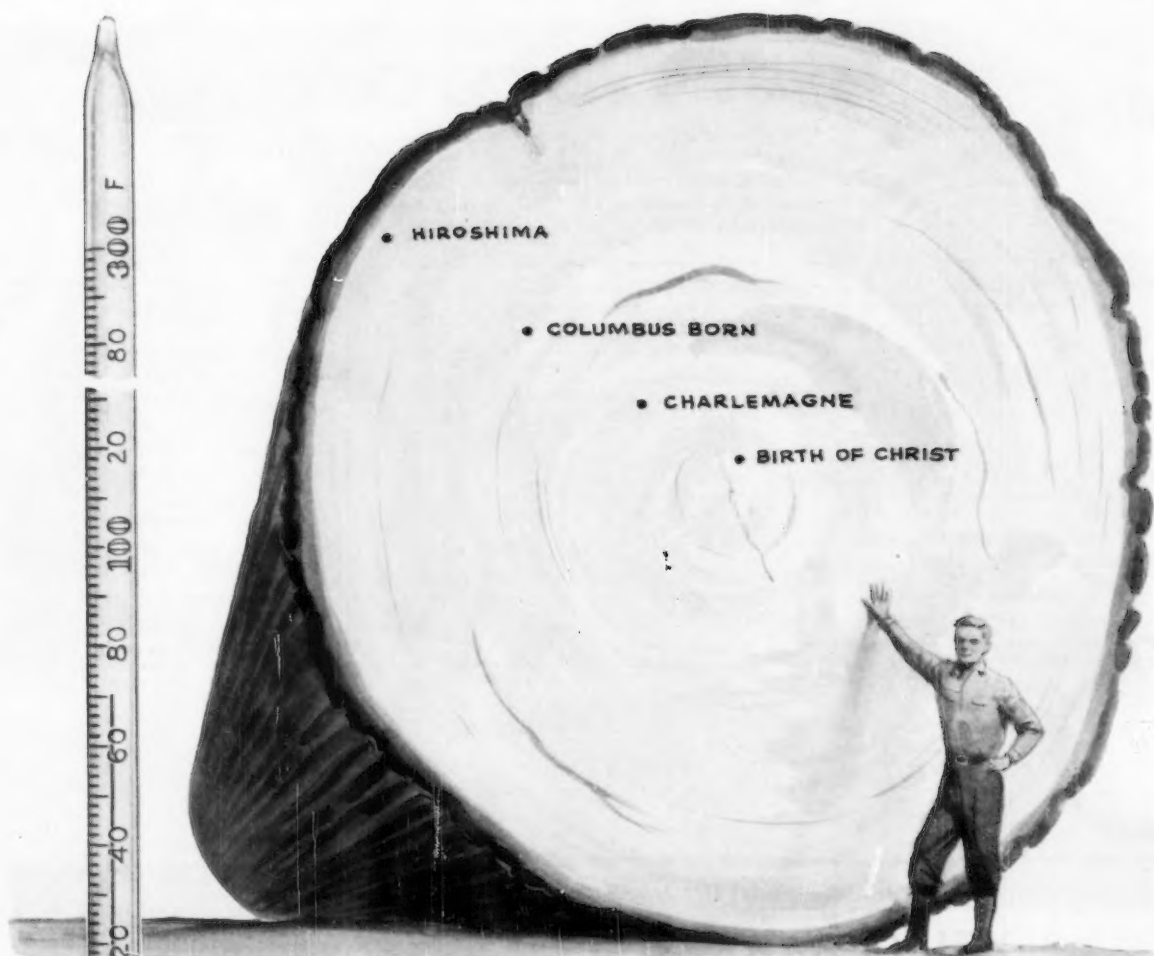
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FOR FURTHER INFORMATION CIRCLE 765 ON READER SERVICE CARD

NEW MEMBERS.....

The following 19 members were elected from November 18 to December 20, 1957, making the total membership 9157 Welcome to ASTM.

Note—Names are arranged alphabetically—company members first then individuals—Your ASTM Year Book shows the areas covered by the respective Districts

CHICAGO DISTRICT (5)

Mitchell, Charles E., soils engineer, Westville Engineering Co., Inc., Box 67, Westville, Ind. For mail: R. R. 2, Box 346, Pear Rd., North Liberty, Ind.
Ward, Don R., president, Engineering Testing Corp., 650 S. Summit Ave., Villa Park, Ill.
White, Edward S., director of research, Warwick Manufacturing Corp., 7300 N. Lehigh Ave., Chicago 31, Ill.

CLEVELAND DISTRICT (4)

Phillips, M. L., Alloys and Chemicals Manufacturing Co., Inc., 4365 Bradley Rd., S. W., Cleveland 9, Ohio.

NEW ENGLAND DISTRICT (13)

O'Connor, Frank B., general superintendent, machine shop, Pattern Shop and Foundry, Electric Boat Div., General Dynamics Corp., Groton, Conn.

Watson, William, research, Craig Systems, Inc., 90 Holten St., Danvers, Mass.

NEW YORK DISTRICT (1)

Orangeburg Manufacturing Co., Emil J. Wall, assistant director of research, Orangeburg, N. Y.
Hescock, Milton A., manager, Paper Pulp Sales, Riordon Sales Corp., Ltd., 220 E. 42nd St., New York 17, N. Y.
Walton, John S. V., resident representative, Marconi's Wireless Telegraph Co., Ltd., 23-25 Beaver St., New York 4, N. Y.

OHIO VALLEY DISTRICT (15)

Max, Abraham M., manager, chemical and physical lab., Engineering Section, RCA Victor Record Div., 501 N. La Salle St., Indianapolis 1, Ind.
Shuler, John H., manager, Engineering Lab., Ranco, Inc., 601 W. Fifth Ave., Columbus 1, Ohio.

SOUTHEAST DISTRICT (17)

Conroy, J. J., engineer, T. L. Herbert and Sons, 174 Third Ave., N., Nashville 3, Tenn.
Yager, T. C., manager, Ready Mixed Concrete Plant, Hardaway Contracting Co., 2001 Taylor St., Columbia, S. C.

WASHINGTON, D. C. DISTRICT (14)

Egan, Richard R., director, applications research, American Alcolac Corp., 3440 Fairfield Rd., Baltimore 26, Md.

U. S. AND POSSESSIONS

Wright, P. L., superintendent, cement plant, Ash Grove Lime and Portland Cement Co., Louisville, Nebr.

OTHER THAN U. S. POSSESSIONS

Marquez P., Edward J., technical director, Suelos Concreto Asfalto, Calle 86A, 8-80, Maracaibo, Venezuela. For mail: Calle 72, 2A-09-Qta Fujiyama, Maracaibo, Venezuela.
Parsons, S. J., managing director, Mersey Cable Works, Ltd., Bootle, Liverpool 20, England.
Sewell, James Fowler, metallurgist, Samuel Fox and Co., Ltd., Stocksbridge Works, Nr. Sheffield, Yorkshire, England.
Vélez Rosas, Raúl, divisional engineer, Industria Eléctrica de México, S. A., Ciudad Eléctrica, Tlalnepantla, Edo. de México.

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GR-5

NEWS NOTES ON Laboratory Supplies and Testing Equipment

Note—This information is based on literature and statements from apparatus manufacturers and laboratory supply houses. The Society is not responsible for statements advanced in this publication.

LABORATORY ITEMS

Error Detector—A combined expanded-scale frequency meter and frequency error detector of unusually rugged and low-cost construction has been developed.

Aerojet-General Corp.

1493

A-C Voltage Regulator—Output voltage of the "Variautoformer" is rectified, filtered and compared to a 0.5 per cent standard voltage derived from a 5651 reference tube. The difference voltage is applied to lead and lag networks; the outputs of the networks are used to control a servo which is mechanically linked to the adjustable arm of the "Variautoformer."

Armour Electronics, Inc.

1494

Insulation Tester—New circuitry of model 250 for accurate resistance measurements from 1 to 50,000 megohms provides outstanding features.

Associated Research, Inc.

1495

High-Output Coil—A special Ateotran differential transformer featuring double range and extremely low null voltage has been developed.

Automatic Timing and Controls, Inc.

1496

Ultrasonic Jack Hammer—Inclusions may be removed from metals and alloys for identification purposes by chemical or mechanical methods. The latter have the advantage that the possibility of chemical change is eliminated. The ultrasonic jack hammer serves for the removal of inclusions of minimum diameter of about 10 microns by means of a pointed stylus oscillating at ultrasonic frequencies.

Brinkmann Instruments, Inc.

1497

Sequence Timer—A sequence timer with switching capacity of 10 amp at 115 vdc or 15 amp at 115 vac with driving motor drawing only 8 ma to 6 vdc is available.

Brailsford & Co., Inc.

1498

Test Oscillator—Specifically designed for repetitive test and maintenance operations in the telecommunications industry, a new test oscillator model 25A features eight pushbutton-selectable frequencies and all-transistor circuitry.

Consolidated Electrodynamics Corp.

1499

Fabric Burn Tester—New tester to determine the resistance to heat of fabrics, upholstery, plastics, etc. is known as the fabric burn hole tester.

Custom Scientific Instruments, Inc.

1500

Transducer Recording—To enable direct recording of the output of differential transformer transducers, a new exciter-demodulator has been introduced. The instrument supplies current regulated ac excitation to the transducer, demodulates and filters the resulting signal and supplies a linear dc output signal proportional to

(Continued on page 88)

THWING-ALBERT ELECTRONIC TENSILE TESTER

An instrument for your present and future testing needs.

Backed by over half a century of leadership and know-how in physical testing instrumentation.

Check these features:

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Sentronic "X-Y" Strip Chart Recorder

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up to 1000 to 1

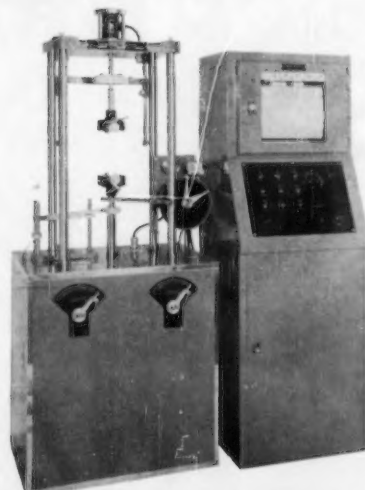
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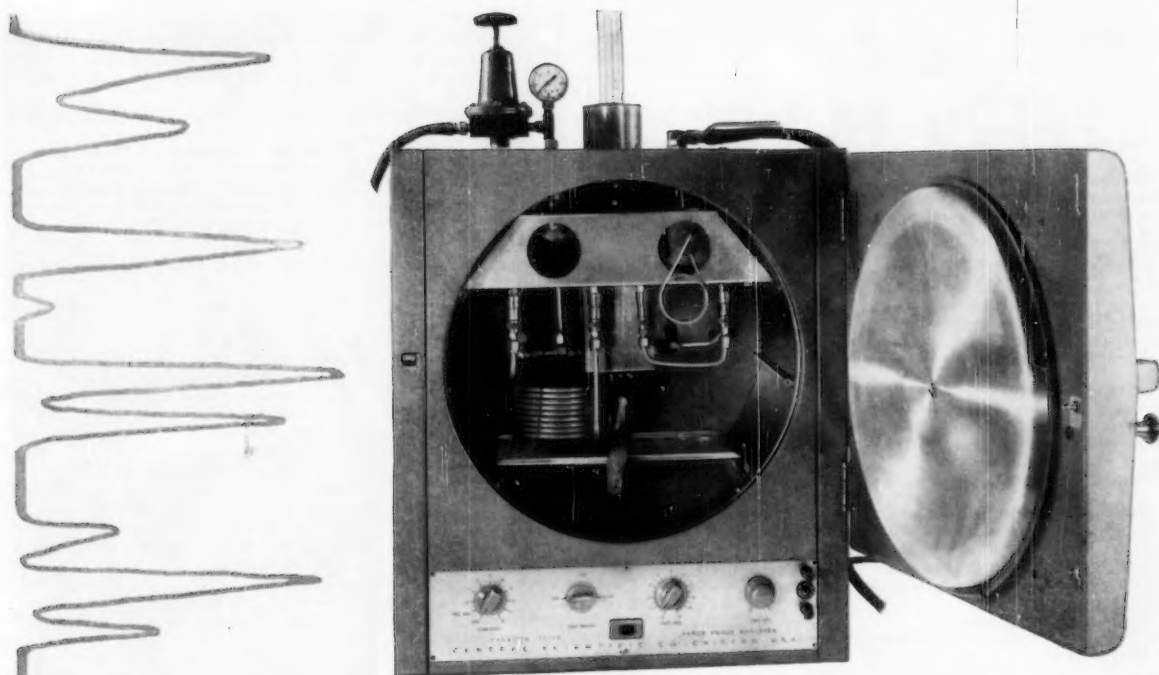
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A most practical instrument of wide temperature range for the analysis of gases or volatile liquids by gas chromatography. Based on the elution technique, the Cenco Analyzer gives a simple reliable method of analyzing chemical and petroleum components quickly and accurately. Built around the popular Cenco constant temperature oven, it provides sensitive control from ambient room temperature to 200°C. Handles liquids which boil over 300°C. Quick-change columns, built-in gas sampling valve and unique liquid sampling feature facilitate analyses. Large variety of column materials available.

No. 70130 Cenco Vapor Phase Analyzer comes equipped with Gow-Mac detection cell, sensitive pressure regulator, flowmeter 1 to 83 ml/min range, 10 ft. coiled column of Di-2-ethylhexyl phthalate, syringe, temperature controls, thermometer, built-in regulated power supply, but without recorder. **\$895.00**

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Lab Items

(Continued from page 86)

transducer input. Direct operation is provided with standard strip chart recorders, X-Y plotters digitizing equipment, etc.

Daytronic Corp. 1501

Mechanical Force Gages—Two new extra-heavy-duty models carry 0-25,000 and 0-50,000 lb capacity ratings.

W. C. Dillon & Co., Inc. 1502

Chlorine Analyzer—Residual chlorine analyzer (model 17A1100) provides continuous automatic measurement of free, combined, or total residual chlorine in water or sewage.

Fischer & Porter Co. 1503

Adhesion Tester—A new instrument provides a means of accurately studying the scratch-hardness versus adhesion properties of organic protective coating systems.

Gardner Laboratory, Inc. 1504

Density Measuring—New density measuring system, model P-625, for use in industry, and for the measurement of the density of liquids during transit, storage, or throughout the complete cycle of a reaction.

General Communication Co. 1505

Mica Capacitors—Type 505 capacitors are available in a 1-2-5 series extending from 100 μ f to 0.5 μ f. Dissipation factor for the 1000- μ f and higher sizes does not

exceed 0.0003. They are housed in low-loss molded-phenolic cases and are equipped with both screw- and lug-type terminals and mounting flanges.

General Radio Co. 1506

Strain Gage Plotter—A new model 220 high-speed strain gage plotter that scans and records four separate channels per second is designed for use with strain gage inputs.

Gilmore Industries, Inc. 1507

Research Microscope—Designed for precise laboratory research, this extremely rigid phase-contrast microscope features a permanently horizontal, mechanical focusing stage with verniers and inclined eye tube for rapid focus control as well as comfortable viewing position.

Karl Heitz, Inc. 1508

High Pressure Valve—The new high-pressure midget valve conserves space and effects economy. The capacity of the system, with a minimum of hold-up is ideal for gage lines, instrument, sampling, and catalytic injection systems.

High Pressure Equipment Co., Inc. 1509

Micro-Microammeter—The model 413 is intended primarily for monitoring rapidly varying currents over a wide range in nuclear reactor control. Its large, single scale reads from 10^{-11} to 10^{-8} amp with no range switching. Both input and output are at ground, making it convenient to connect recorders.

Keithley Instruments, Inc. 1510

Generator—"Atmo-Carb" controlled Atmosphere Electric Furnaces, are designed for the heat-treating of steel for labs.

L & L Mfg. Co. 1511

Portable Fluorescent—A completely portable fluorescent penetrant inspection kit is available for locating cracks, pores, leaks, and other defects open to the surface in any solid material. This kit is known as the Zyglo ZA-43.

Magnaflux Corp. 1512

Vacuum Furnace—A versatile new laboratory vacuum furnace, designated the F-15, is built on the cost-saving modular principle for maximum adaptability to many different high vacuum metallurgical research applications.

The New York Air Brake Co. 1513
Kinney Mfg. Div.

Pulse-Height Analyzer and Scaler—Model 132 analyzer computer, designed for use with all gamma-sensitive scintillation counters combines a well regulated high-voltage supply, single channel pulse-height analyzer, binary scaler, and an automatic push-button computing circuit, all in one chassis.

Nuclear-Chicago Corp. 1514

Scaler—A new universal scaler featuring a wide choice of optional slide-out

(Continued on page 92)


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FOR THE MOST RAPID MOUNTING OF METALLURGICAL SAMPLES

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The most versatile and accurate instrument yet designed, this Marconi Model TF-933A Moisture Meter has been developed and exhaustively tested in England for over three years, and is now available to American industry.

Excellent results may be obtained on most organic substances, with an accuracy of $\pm 0.2\%$ under controlled conditions. No special skills are required and precise measurement takes less than a minute. Supplied with temperature compensation scales. Range for pulp 6-22%—for paper 4-13% with special electrode available to extend range down to 2% and also sword type electrodes for inner stack measurements. Chart calibrations also available for 150 other substances such as grains, tobacco, flour, etc. Immediate delivery from New York stock, with service and spares available.

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January 1958

John SPRAY BOTTLE for CHROMATOGRAPHY

Uniformly fine spray
Instantaneous control
For wide variety of
liquids



9186-R2.

SPRAY BOTTLE, CHROMATOGRAPHIC (ATOMIZER), John (Patent pending). An ingenious spray tube provides uniformly fine mist or spray, as required for paper chromatography, from a wide variety of liquids with high or low viscosities, different vapor pressures, etc. Sprays large areas conveniently from a distance of approximately three feet when connected with a source of air pressure of three lbs. per square inch, but a pressure of five lbs. per square inch is recommended.

Instantaneous control of spray is provided by applying thumb to a $\frac{3}{8}$ -inch vent in rear of the spray tube. Spray tube is of Pyrex brand glass, with permanently attached plastic screw cap size No. 24 which fits commercial narrow mouth, screw neck bottles. Overall height $7\frac{1}{2}$ inches; takes rubber tubing $\frac{1}{4}$ -inch bore.

9186-R2. Spray Bottle, Chromatographic (Atomizer), John (Patent pending), as above described, with spray tube, screw cap and 8 oz. glass bottle, but without air pump or rubber tubing . . . 8.00

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SCOTT TESTER* MODEL LG

Test Tube Ageing up to 550°F

This modern insulated solid aluminum block electric oven tests elastomers at precise temperatures, positively prevents contamination between samples in the 28 isolated compartments.

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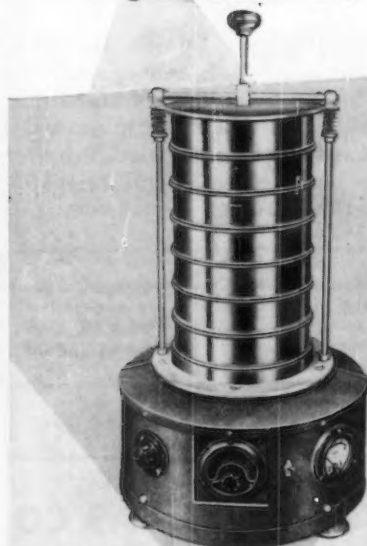
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FOR FURTHER INFORMATION CIRCLE 774 ON READER SERVICE CARD

SYNTRON TEST SIEVE SHAKERS

*for faster, more accurate
analysis of industrial materials*



**NEW,
IMPROVED**

TS158

The new, improved SYNTRON Test Sieve Shakers provide quick accurate separation of industrial materials with more positive control of components.

Featured improvements

- Larger, more powerful electromagnetic drive
- Rheostat voltage control
- Reset timer for accurate time testing
- Voltmeter to obtain comparable tests under identical conditions
- Holds six standard 8-inch test sieves and bottom pan
- Operates from 115 volt, 60 cycle a-c.

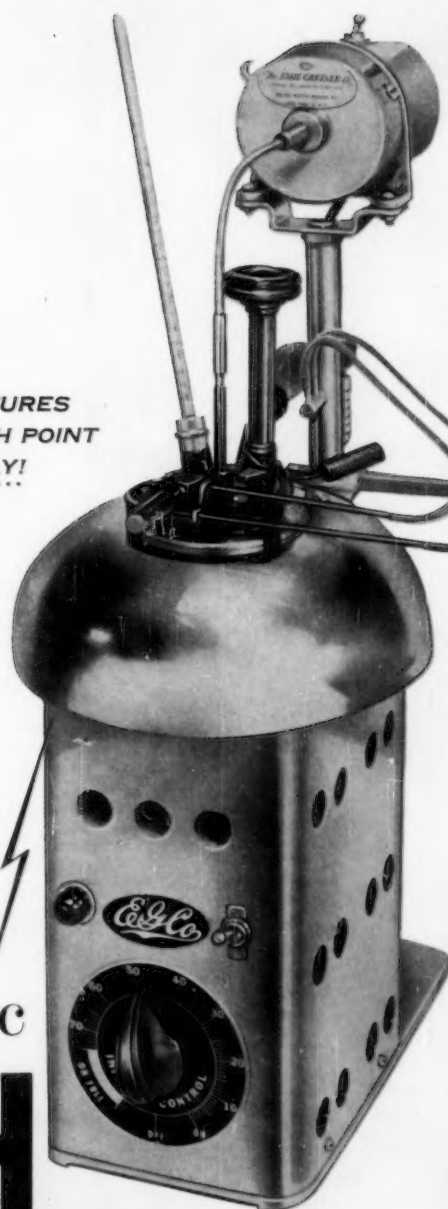
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BRING TEMPERATURES
UP TO FLASH POINT
AUTOMATICALLY!

new
infinite
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electric

FLASH TESTERS



With a single setting of the dial, new infinite control automatically increases the temperature at a specified rate within the test range. Pointer setting determines number of seconds the heater will be on, as indicated by the pilot light.

PENSKY MARTENS

CLOSED TESTER A.S.T.M. D93

- Temperature increases 9° to 11° F per minute in range of flash point.
- New short flexible shaft on stirrer is positive and easily disconnected.
- New coil for rapid air cooling—perforated tube around the iron casting has a serrated connection to air line.
- New locking device holds cover and cup together... is quickly disconnected... insures safer operation.
- New stainless steel shell and casing mounted on aluminum base casting.
- 750 W, 110 V AC heater

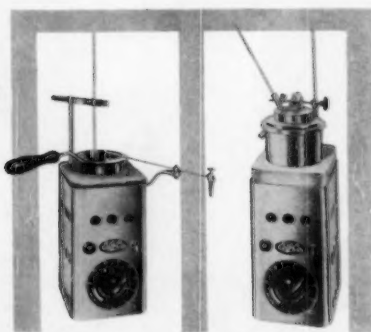
G-18196

Complete with motor stirrer—195.00

TAG CLOSED TESTER A.S.T.M. D56

- Temperature increases 2° F per minute, as specified in method.
- 150 W, 110 V AC heater

G-18181N 110.00



G-18081

G-18181N

CLEVELAND OPEN CUP FLASH TESTER A.S.T.M. D92

- Temperature increases 9° to 11° F per minute in range of flash point.
- New, easily replaceable heating unit.
- Thermometer swings back out of way when not in use.
- 750 W, 110 V AC heater.

(B)

G-18081 125.00



The EMIL GREINER Co.

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FOR FURTHER INFORMATION CIRCLE 776 ON READER SERVICE CARD



Series D Gages are made in 5 models for measuring load ranges from 0-50 pounds to 0-200 pounds.

The Force Gages specified in many test "standards"

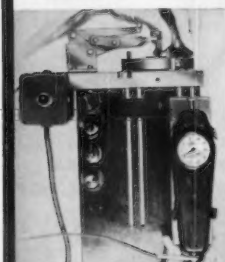
PRECISION FORCE GAGES



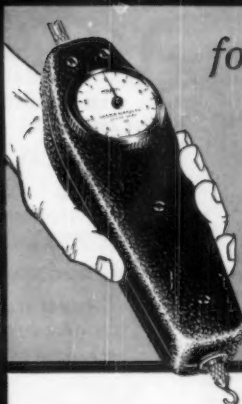
In laboratory, testing bond strength of foil coatings.



In field, as tool for adjusting aircraft controls.



On production line, determining carbon brush pressure.



for measuring tension and compression loads

Series L Gages are made in 13 models for measuring load ranges from 0-500 grams to 0-30 pounds.

Hunter Mechanical Force Gages are precision, direct reading instruments designed for use in inspection departments and laboratories, on production lines, or in the field to measure tension and compression loads. Accurate to within 1% of full scale, they can be hand-held or used in fixtures depending upon use.

The indicator can be made to hold maximum reading until released (no "follower" hand used) or the indicator can be made free moving. Gages are available as noted under illustrations above. Each unit is supplied with six attachments—extension rod, flat knob, notch, hook, cone, and chisel edge—in a compact wood instrument box. Prices are low enough to permit any company to buy one for evaluation. Quantity prices are also offered.

Hunter Bulletin 750c contains full details. Copies are available immediately on request.



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Manufacturers of: Precision Springs, NEG'ATOR® & FLEX'ATOR® Constant-Force Springs, Stampings & Assemblies, Retriever Reels, Force Gages, and Wire Fatigue Testers

FOR FURTHER INFORMATION CIRCLE 777 ON READER SERVICE CARD

Lab Items

(Continued from page 88)

plug-in sections allows a customer to order an instrument built to his own needs.

Nucleonic Corp. of America 1515

Electrometer—Model 33B electrometer is a stable instrument for the measurement of small d-c voltages and currents, derived from high impedance sources.

Robertshaw-Fulton Controls Co. 1516

Miniature Load Cells—New miniature low-capacity differential transformer type load cells for measuring weight, force, twist, or torque.

Testing Equipment Sales Co. 1517

Temperature Scanner—Versatile multi-point temperature scanner provides over-temperature or under-temperature protection for systems where it is mechanically or economically desirable to provide one temperature alarm instrument capable of sensing several control points and providing alarm if one of these temperatures exceeds or fails to reach a pre-set limit.

Tiptronic, Inc. 1518

High-Vacuum Pump—Known as the VS-9, the fully equipped unit provides industry and research laboratories with multipurpose high-vacuum station incorporating these features: very fast pump-down; extremely high vacuum—with bakeout, better than 10^{-6} mm Hg; and a high speed air-cooled diffusion pump with an 85 liter per sec capacity.

Veeco Vacuum Corp. 1519

CATALOGS AND LITERATURE

Refractometer—A four-page Bulletin, No. 2290, describes the Aminco-Jonnard refractometer, a new instrument which measures refractive index differences to an accuracy of 0.00002.

American Instrument Co., Inc. 2333

Adsorbents—A 4-page Bulletin, No. 855, is a guide and price list of packaged adsorbents for use in all chromatographic instruments.

Burrell Corp. 2334

Tintometer—An eight-page catalog, No. T17, describes tintometers, applications, and technical data given.

Curry & Paxton, Inc. 2335

Force Control Switches—An illustrated bulletin describes a complete line of force control switches, designed for controlling mechanical forces electrically.

W. C. Dillon & Co., Inc. 2336

Magnetic Flowmeter—Catalog 10D1416 discusses principle and operation of magnetic flowmeters; it gives advantages and specifications.

Fischer & Porter Co. 2337

Burets—Catalog 89-24 describes new line of Fischer & Porter Accurette precision-bore burets fashioned of special tubing with bore tolerances of ± 0.0002 . Burets meet or exceed all standards set forth in National Bureau of Standards, Circular C434.

Fischer & Porter Co. 2338

Stress Rupture—Complete information about temperature control equipment for stress-rupture and creep testing is available in a new 2-page data sheet 247(1).

Leeds & Northrup Co. 2339

(Continued on page 94)

HARSHAW SCIENTIFIC

SPOTLIGHTS Centrifuges

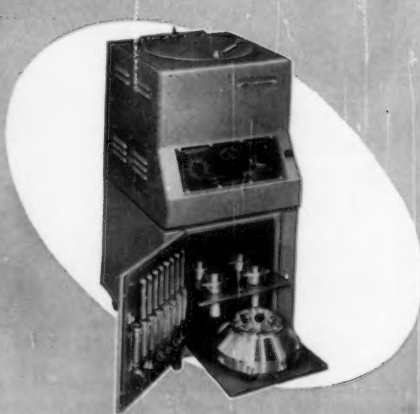
Two new centrifuges have been added to our already extensive line of International centrifuges. The new HT for high speed centrifuging and the new CM, a cabinet unit, for routine moderate speed work. The HT high speed centrifuge provides speeds to 17,000 r.p.m., with forces to 34,390 x G and is designed for maximum safety to operating personnel. The CM centrifuge features great adaptability at moderate price. This attractively designed

unit accommodates 23 heads and accessories for any routine application.

An International centrifuge is available for every need from stock. The complete line includes micro, clinical, chemical, routine, large capacity, explosion proof, blood plasma, refrigerated, oil testing, soil testing, and a number of other special application centrifuges. Complete information is available on the centrifuge you need. Write for literature.



H-9985—International Model HT, with steel guard bowl, complete instrumentation and 8-place angle head for 50 ml tubes (without tubes). For operation on 115 volts, 50/60 cycles, single phase \$745.00



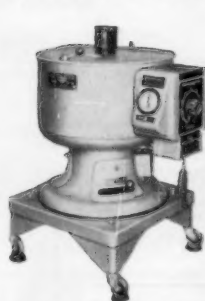
H-8800—International Size 1, Model CM, with stainless steel guard bowl, autotransformer controller, electric tachometer and timer but without head, shields or cabinet floor stand; 115 volts, 50/60 cycles \$475.00



H-8500—
Clinical Model



H-8858—Size 1, SBV
Routine and Research



H-9506—Size 2, V
Large Capacity, High Speed



H-9875—Model PR-2
Refrigerated

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FOR INFORMATION



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Hastings-on-Hudson 6, N. Y.

FOR FURTHER INFORMATION CIRCLE 778 ON READER SERVICE CARD

Catalogs

(Continued from page 92)

High Frequency Induction—A 12-page catalog, *Bulletin No. T-1067*, fully describes the complete line of high-frequency induction units and accessories for rapid high-temperature combustion of laboratory samples.

Lindberg Engineering Co. 2340

Electronic Densitometer—Technical details and step by step photos are presented in this new bulletin which gives the detailed story of the new Macbeth-Ansco densitometer model 12-A.

Macbeth Daylighting Corp. 2341

Thermal Element—Selection of thermal elements for maximum resistance to corrosive atmospheres is made easy with a new folder, *Bulletin No. 110*.

The Parlow Corp. 2342

Radiation Counter—A four-color, 132-page catalog contains the manufacturer's complete line of 200 items, ranging from Geiger Mueller detectors to transistorized multi-channel analyzers.

Radiation Counter Labs., Inc. 2343

Portable Potentiometer—New 6-page bulletin, *No. T-57*, features the thermo-

stat portable potentiometer for measuring temperatures and voltages.

Technique Associates, Inc. 2344

Laboratory Weights—A 22-page bulletin, *No. W-57*, describes construction and tolerance details for laboratory weights.

Henry Troemner, Inc. 2345

INSTRUMENT COMPANY NEWS

Labasco, Inc., Durham, N. C.—Under the corporate name of LABASCO, Inc. a new nationwide service organization has been formed to furnish laboratory instruments and supplies to industrial, educational, and government laboratories. The Labasco affiliates are: Cardinal Products, Inc., Chemical Rubber Co., Industrial Scientific Co., Macalaster-Bicknell Co., B. Preiser Co., Schaar & Co., and Standard Scientific Supply Corp.

Macalaster-Bicknell Co., Cambridge, Mass.—Manufacturers and distributors of laboratory and industrial supplies, has opened a new glass plant in Fitzwilliam, N. H., near Keene. It will be devoted entirely to producing glassware for research and industry.

NEWS OF LABORATORIES

United Electrodynamics, Pasadena, Calif.—Announcement was made by United ElectroDynamics, of this city, of the opening of a new facility to be called the United Testing Laboratories. This move represents a separation and expansion of the research and commercial testing operations carried on by the company for a number of years.

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*You'll need
this useful
16-page Booklet*

The 16-page bulletin illustrated above catalogs the apparatus and instruments you'll need in your laboratory for metallographic analysis. It shows equipment which will help you improve the quality of your analyses, help you do them in less time and

with more efficiency. You'll find this bulletin particularly useful if you are up-dating your present laboratory or designing a new one.

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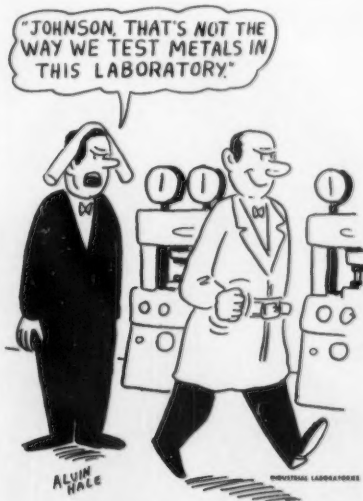


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TESTING MACHINE DIVISION

AMSLER HIGH FREQUENCY VIBROPHORES

The Amsler High Frequency Vibrophore is one of the latest products of this world-renowned manufacturer of physical testing equipment.

It determines the fatigue properties of practically any material under fluctuating and alternating tensile, compressive, shear or torsional stresses.

Simple specimens or test bars, sections of assembled structures or machine components can be tested.

Two sizes of machines cover the load range from 44 to 22,000 lbs. The Vibrophore uses the classic principle of resonance which permits frequencies up to 300 cycles per second while ensuring the accurate maintenance of stresses within 1% of magnitude and time constancy.

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Random Samples

(Continued from page 27)

Floated, it disperses slowly to form a film. The initial charge of about 8 lb (at 65 cents a pound) is expected to last about two weeks in the Rattlesnake Reservoir tests.

Each molecule is only a fraction of a millionth of an inch long. The principle is that the molecules will compress as a monolayer, intervening between the water and the air and sun that steal away molecules of water.

Hexadecanol is a commonplace ingredient of certain medicinals, cosmetics, and other commercial products. In reservoir use, of course, the amounts of the chemical used are literally infinitesimal in comparison to the millions of gallons of water stored. In experimental use at Kid's Lake near Oklahoma City during the summer of 1956, it had no discernible effects on aquatic life, was completely nontoxic to higher animals, and did not affect the taste and odor of the water.

The studies of techniques at the Colorado sites by the Bureau of Reclamation are preliminary to full-scale monomolecular layer experiments sched-

uled to be held this summer at Lake Hefner, the main reservoir for Oklahoma City, if the results of the tests currently under way at the Colorado reservoirs turn out to be favorable. There, attempts are to be made actually to measure the reduction of evaporation losses.

Drought in the West has focused not only public attention, but also the attention of science on various means of augmenting scant water supplies. Research into use of hexadecanol has been active in Australia and South Africa for several years, and the Southwest Research Inst. is working along similar lines in Texas.

Chemical News, Manufacturing Chemists Assn., July-August, 1957.

Thorium Declassified

LONG kept in a "top secret" file, the full story on the development of thorium metal has been declassified by the Atomic Energy Commission.

In 1951, Government research workers found that thorium could produce U-233, which, in turn, had fissionable properties. It was first believed that

these materials would develop greater energy, gram for gram, than uranium-based derivatives.

What was needed to test these theories was a way to produce a high-purity thorium metal at a lesser cost. Until that time, the most effective method of recovering thorium was by the Ames process, reacting thorium tetrafluoride with calcium metal in the presence of zinc chloride. This formed a thorium zinc alloy which was then treated in a vacuum to distill off the zinc. The resultant thorium is described as "moderately pure," with a production cost of between \$15 and \$20 per lb.

In April, 1952, the search for a cheaper and purer thorium was launched by Horizons, Inc., Cleveland, under a research contract from the Atomic Energy commission. Horizons' metallurgists, with broad experience in the recovery of metals through the use of fused salt electrolysis, sought a way to apply the same process to the extraction of thorium. The final step, the actual electrolysis which deposits thorium on a cathode, was not actually the big problem. The big research job was to find an electrolyte or cell feed material which would easily yield very high-purity thorium.

AEC specifications dictated that the thorium recovered must be about 99.9 per cent pure. Two years of exacting research work followed.

The problem of developing a proper cell feed was not easily overcome. Laboratory standards were extremely stringent. For example, initially, in batch after batch of thorium developed, spectroscopic examination would reveal 15 to 20 parts of boron per million parts of thorium. AEC specifications demanded that there be less than 1 part boron per million parts thorium. No one knew why boron was occurring in such comparatively heavy quantities until a technician one day noticed that a number of the workmen were using a borax preparation to wash their hands before handling the laboratory and pilot plant apparatus, which later showed up in the spectroscopic. Substitution of soap for borax powder eliminated the problem.

By 1954, Horizons research had developed a cell feed which, through the fused electrolysis process, would yield thorium to the purity specifications of AEC, at a conversion cost of approximately \$2 per lb.

The process involves reacting thorium nitrate (the base material) with sodium carbonate to recover thorium oxycarbonate, sodium nitrate, and carbon dioxide. The thorium oxycarbonate precipitates out as white solid. This selective precipitation in the first step

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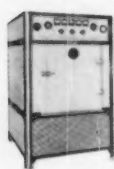
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(Continued on page 102)

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The delivery unit contains a valve mechanism with an adjustable rate controller, an open ended motor driven chuck for the stirrer, stirring motor and electrode holders. The valve mechanism,

mounted on a support with swinging beaker platform and a clamp for the titrating burette, is designed to accommodate any form of burette with or without stopcocks, so that it may apply to any type of titrating installation.

Dimensions of control unit: height, 7 $\frac{3}{4}$ inches; width, 10 inches; depth, 9 inches; power consumption, 100 watts; total shipping weight, 35 lbs.

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The Bookshelf

Underground Corrosion

Melvin Romanoff, National Bureau of Standards Circular 579, U. S. Government Printing Office, Washington (1957), 227 pp., \$3.

THE corrosion of metallic structures buried in soils or in contact with soils has long been a serious engineering and economic problem. It is the aim of this new Circular to provide a useful reference for the technician who is interested in the theoretical aspects of underground corrosion and a practical reference for the engineer who may be interested only in the methods commonly used to prevent corrosion.

This Circular supersedes NBS Circular 450 issued in 1945 and is a final report on the studies of underground corrosion conducted by the National Bureau of Standards from 1910 until 1955. It contains a condensed summary of the Bureau investigation on the corrosion of metals in soils conducted over a period of 45 years. There are 114 tables and 103 graphs and illustrations. Results of both field and laboratory investigations, utilizing more than 36,500 specimens of a wide variety of ferrous and non-ferrous materials exposed in 128 test locations throughout the United States, are presented.

Neutron and Gamma Irradiation Facilities

Atomic Energy Commission, Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C., 60 cents.

ONE of the big problems facing those interested in experimentation is obtaining data on the facilities available for irradiating materials. This volume was prepared specially for engineers and scientists engaged in or contemplating irradiation research or development of industrial applications of irradiation. The book is divided into two parts: Reactor Irradiation Facilities and Gamma Irradiation Facilities. Part 1 describes the power and type of the reactors, status (whether or not currently operating), fast neutron flux, gamma flux, core description, moderator, reflector, coolant, and shield, and operating schedules. Part 2 gives such details of gamma irradiation facilities as maximum flux, type of source, sample environment (water, air, or both) for each facility, and operating procedures.

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Radiography in Modern Industry

Eastman Kodak Co., X-Ray Division, Rochester 4, N. Y., \$5.

THIS is a practical treatise on all aspects of industrial radiography. It should be of inestimable value to the X-ray technician and could readily be used either as a textbook in classrooms or as a handbook for actual practice.

The book consists of 140 pages with over 100 excellent text illustrations. A bibliography and cross index are included.

OTS Research Reports

These reports, recently made available to the public, can be obtained from the Office of Technical Services, U. S. Department of Commerce, Washington 25, D. C. Order by number.

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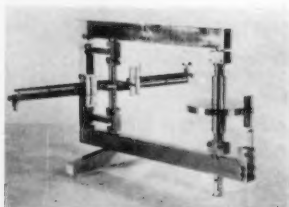
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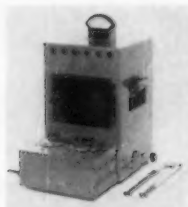
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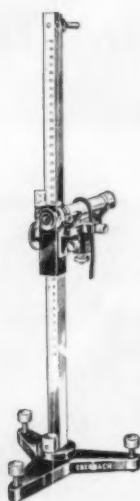
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January 1958

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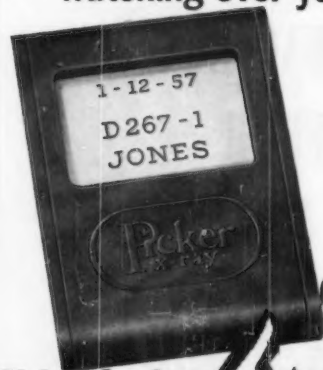
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Random Samples

(Continued from page 96)

eliminates many of the impurities which could occur later on. The thorium oxide (which can be derived from thorium oxycarbonate by heating) is then dispersed on finely powdered lamp black, this mixture being treated with chlorine at a temperature of between 1350 and 1400 F. This process yields thorium tetrachloride, along with carbon monoxide and carbon dioxide. The thorium tetrachloride is then fused at about the same temperature with sodium chloride in the proportion of 70 per cent thorium tetrachloride, 30 per cent sodium chloride. This substance is used as the electrolyte in the final recovery step. The fused salt, with all the properties of a liquid, is placed in a graphite-lined cell, with the graphite being a positive electrical post, and a removable cathode being the negative post. Thorium salts are deposited on the negative cathode, which is removed as soon as the accumulation is heavy enough to warrant removal. The entire reaction takes place in an inert argon atmosphere, to prevent rapid oxidation of the materials involved. Amperages up to 7500 have been used in the process.

By April 1954, the work had moved from laboratory type operations to a production type facility of the Atomic Energy Commission. While the original suppositions that thorium's energy content would supersede that of uranium have not proven correct, the greatly reduced cost of production provided by Horizons' work, and the reduced density of thorium as against uranium give the material a strong future potential. One firm, under contract to Brookhaven National Laboratory, is now in the process of constructing a liquid metal fuel homogeneous reactor, which employs at least in part of its operations a thorium-bismuthide slurry as the reactor fuel.

Because it is less dense (and thus lighter) than uranium, thorium may prove extremely useful in reactors of the future for atomic flight.

Flux Barrier Broken

BELL Telephone Laboratories' new aluminum soldering process has broken the "flux barrier." Flux and vigorous abrasion are no longer needed to bond aluminum, Bell says. And its solder will wet the surface of the metal even though rolling mill oils and the surface oxide have not been removed.

The solder is made chiefly of zinc and can be alloyed with small amounts of aluminum and magnesium. The zinc is kept pure. Lead, tin, bismuth,

and cadmium are kept out of the zinc to prevent intergranular corrosion.

A single stroke of the solder stick across the aluminum surface, which has been treated electrically or with a torch, penetrates the oxide coating and wets the aluminum. The oxide film is lifted off like paint peeling from wet wood. The oxide coating can then be wiped aside.

Thus wet, surfaces can be joined by bringing them together while they're hot and drawing the solder stick across the joint. The joints are stronger than the aluminum itself, Bell claims. The process is most effective with those joints which are accessible for working with the solder stick, such as butt and "T" joints. Besides bonding aluminum, the process is equally effective for joining galvanized surfaces without a flux.

The soldering techniques were developed by G. M. Bouton and P. R. White, metallurgists at Bell Laboratories.

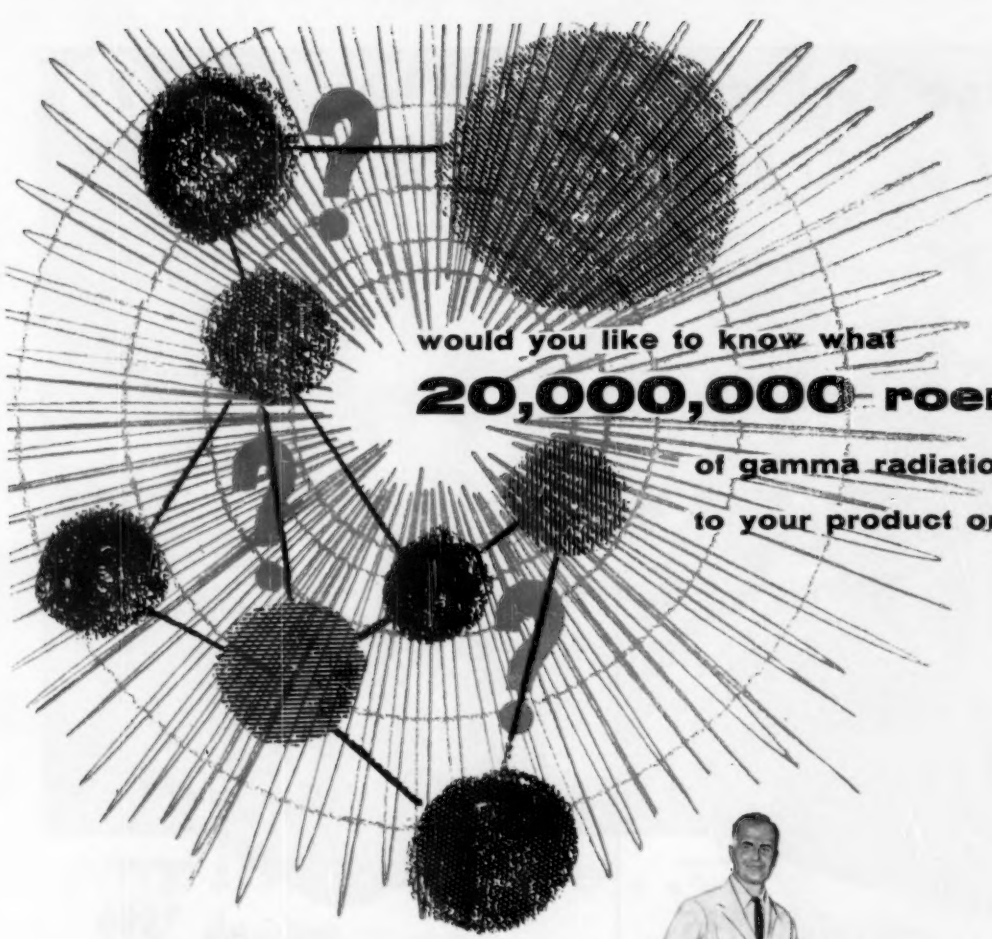
Chemical and Engineering News,

October 28, 1957.

Rheology Society's Medalist Stresses Importance of Theoretical Models in Research

A TIMELY plea for the theoretical and the interdependence of the theorist and the experimentalist was made by Clarence M. Zener, director of the research laboratories of Westinghouse Electric Corp., upon receipt of the Bingham Medal of the Society of Rheology at its annual meeting on November 7. In his acceptance speech Dr. Zener stressed the importance of theoretical models in scientific work, pointing out that they not only provide powerful generalizations which serve to unify many observed phenomena into a few relatively simple concepts but they also provide a basis for future experiments, the results of which may either confirm the validity of a suggested model or suggest necessary modifications of it.

The three-day meeting of the Society, held at the Textile Research Institute in Princeton, N. J., showed a record attendance. J. H. Dillon of the Textile Research Inst. was elected president; J. H. Elliott, Hercules Powder Co., vice-president; W. R. Willets, Titanium Pigment Corp., secretary-treasurer; R. D. Andrews, Dow Chemical Co., editor. R. S. Marvin, National Bureau of Standards, and R. S. Rivlin, Brown University, were elected to the executive committee which also includes the officers and the immediate past-president. Mr. Willets has long been an active member of ASTM.



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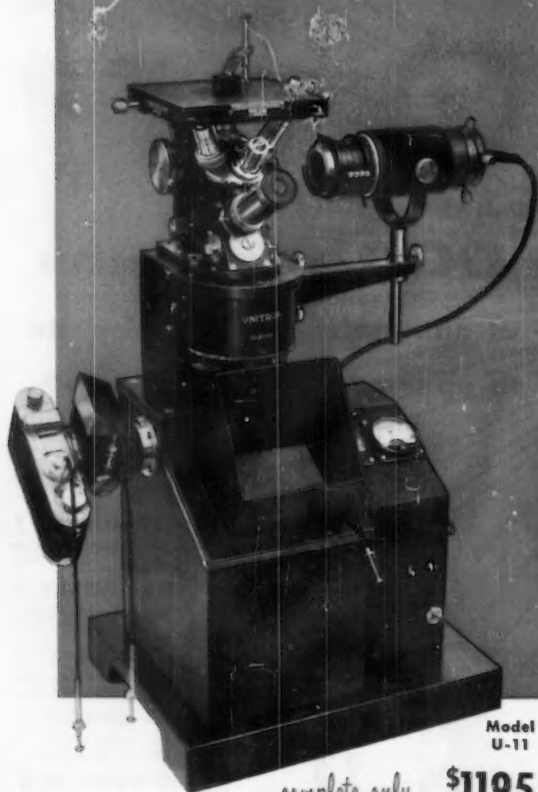
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- ▶ Standard optics include 4 parafocal objective lenses: 5X, 10X, 40X, 100X oil immersion on revolving nosepiece; 3 eyepieces: P5X, Micrometer 10X, K15X, all coated. Magnification range 25-1500X.
- ▶ Vertical illuminator with iris diaphragm. Transformer housed in microscope base. A microswitch on the base provides an extra high intensity for photography.
- ▶ Binocular model has provision for attaching 35mm camera to microscope base. A 35mm camera attachment is available to attach to the eyepiece tube of the monocular model.
- ▶ Calibrated square mechanical stage with calibrated rotatable stage plate.
- ▶ Calibrated polarizing apparatus, 5 filters, dustcover, cabinet, etc. all included.
- ▶ Additional accessories available at extra cost include: 35mm camera attachment; K20X eyepiece for 2000X; transmitted-light accessories for transparent specimens; vacuum heating stage.



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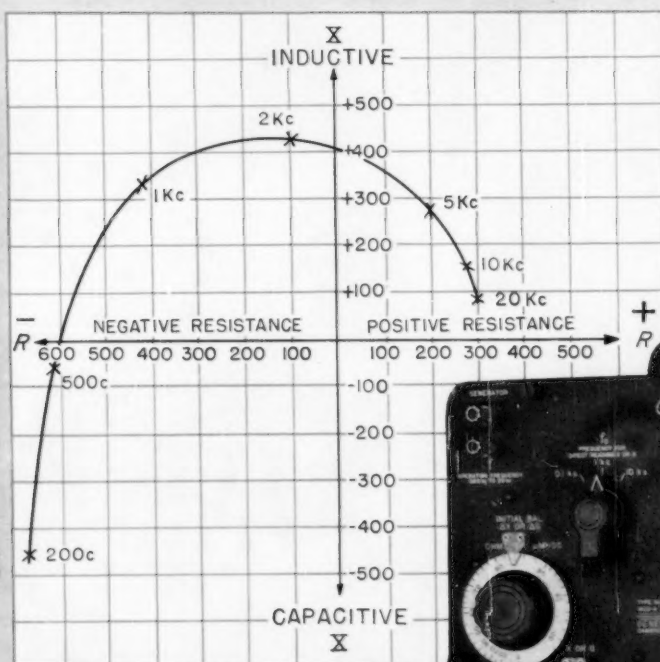
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Impedance of Feedback Circuit . . . illustrates ability of the Z-Y Bridge to measure any impedance: quadrature components may be positive or negative, real or imaginary.



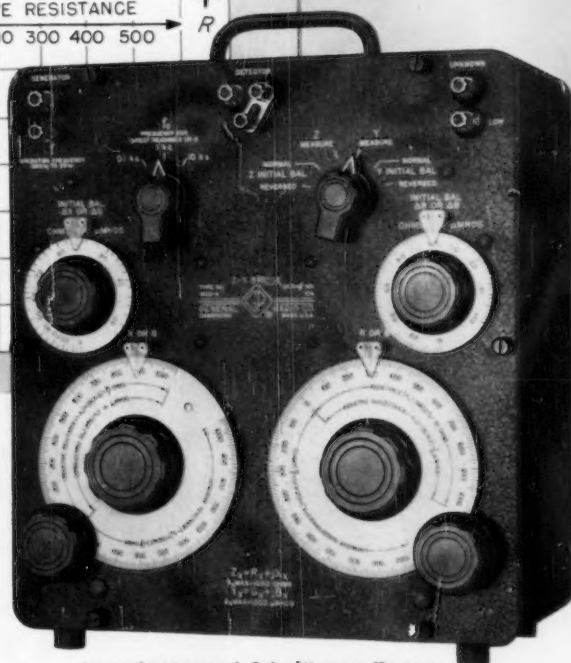
Measures Any Impedance . . .

- ... From 0 to ∞ ohms
- ... Positive or Negative
- ... At Any Phase Angle
- ... Over 20-cps to 20-kc Range

The General Radio Z-Y Bridge measures impedances from short circuit to open circuit, at small or large phase angle. Quadrature components, R & X or G & B, are measured directly at calibrated 100c, 1-kc, and 10-kc bridge positions. Basic accuracy is 1% over most of this range.

The ability to measure impedances of any magnitude accurately with one instrument is an extremely valuable asset in many measurement situations. The Z-Y Bridge can be used for measuring conductivity of liquids in dielectric cells as readily as it can be used for R-L-C component measurements in the laboratory or production-test department. It will measure open- and short-circuit transformer parameters . . . impedances of batteries and electrolytic capacitors . . . characteristics of audio-transmission networks . . . impedance of electro-acoustic transducers . . . Q and resonant frequency of chokes . . . and impedances of feedback loops, since negative real parameters can be directly measured.

The Bridge also can be used to determine cable-fault locations and circular-arc plots of liquids or solids having lossy polarizations in the audio-frequency range. These are but a few of the countless applications for this unique and versatile device. *You name it — this Z-Y Bridge can probably measure it.*



Impedance and Admittance Range
R: ± 1000 ohms G: ± 1000 μ mhos
X: ± 1000 ohms B: ± 1000 μ mhos

Accuracy
R or G: $\pm(1\% + (2 \text{ ohm or } 2 \mu\text{mho}))$
X or B: $\pm(1\% + (2f_o \text{ ohm or } 2f \mu\text{mho}))$

f is operating frequency, f_o is frequency setting of panel selector switch

Impedances of less than 100 Ω (or 100 μ mhos) can be measured on "Initial Balance" dials with considerably greater accuracy —
R or G: $\pm(1\% + (0.2 \text{ ohm or } 0.2 \mu\text{mho}))$
X or B: $\pm(1\% + (0.2f_o \text{ ohm or } 0.2f \mu\text{mho}))$

Frequency Range — 20 cycles to 20 kc

Maximum Applied Voltage
130 volts, rms on bridge;
less than 32v on unknown

Accessories Recommended
Type 1210-B Unit R-C Oscillator and
Type 1212-A Unit Null Detector

Accessories Supplied
2 Shielded Cables for generator and detector

Dimensions — 12½" x 13½" x 8½"

Net Weight — 21½ lbs.

Type 1603-A Z-Y Bridge \$370

GENERAL RADIO Company

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